



NRL Report 8092

## Carbon-13 and Proton NMR Spectra for Characterizing Thermosetting Polymer Systems

I: Epoxy Resins and Curing Agents

C. F. Poranski, Jr., W. B. Moniz, D. L. Birkle, J. T. Kopfle, and S. A. Sojka

Organic Chemistry Branch Chemistry Division

June 20, 1977

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#### CONTENTS

INTRODUCTION	1
SCOPE	1
FORMAT	2
CAUTION	2
EXPERIMENTAL PROCEDURES	2
OTHER NMR SPECTRA CATALOGS	3
~ .	3
REFERENCES	1
APPENDIX A — Carbon-13 NMR Line Listings for Three Materials	
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### CARBON-13 AND PROTON NMR SPECTRA FOR CHARACTERIZING THERMOSETTING POLYMER SYSTEMS 1. EPOXY RESINS AND CURING AGENTS

#### INTRODUCTION

High-resolution nuclear magnetic resonance (NMR) spectroscopy has become a valuable technique for the analysis of molecular structure of organic materials. The nuclei most studied are <sup>13</sup>C and hydrogen (proton). Recent advances in instrumentation have made both <sup>13</sup>C and proton NMR rapid and reliable. In addition, NMR, especially <sup>13</sup>C NMR, is much more specific than other spectroscopic techniques, such as infrared spectroscopy. Often, mixtures can be analyzed by inspection of the spectrum of the unseparated sample. Nuclear magnetic resonance has been widely employed in the study of thermoslatic polymers [1,2], but has been employed much less in the study of thermosetting polymers. New pulse NMR techniques are now making it possible to study solid polymers [3,4].

This report is a catalog that resulted from our need for reference proton and  $^{13}\mathrm{C}$  NMR spectra for use in characterizing epoxy resin systems [5]. Data on a few of these materials are scattered throughout the chemical literature. However, we feel that this collection of both proton and  $^{13}\mathrm{C}$  NMR spectra of epoxy resins and curing agents will be useful to others working in polymer characterization.

Because this catalog is designed for the practicing NMR spectroscopist, we have not presented any discussion of NMR theory or practice. However, a number of references [6-10] are included for the guidance of those in the polymer analysis field who are unfamiliar with NMR techniques.

#### SCOPE

This volume is restricted to epoxy resins and curing agents. A future volume will include plasticizers, more complex epoxy systems, and precursors (or prepolymers) of other thermosetting polymers such as polyimides.

We have not included the spectra of all the epoxy resins that we have investigated. In some cases, spectra for materials of the same viscosity range from different manufacturers were so similar that the inclusion of multiple spectra would serve no useful purpose. However, those cases are noted in the index of spectra that appears in Appendix A.

#### **FORMAT**

With the <sup>13</sup>C spectra the following information for each material is given: chemical name or chemical nature, trade name or other source, solvent used, structure, and table of chemical shifts. With the proton spectra only the name, source, and solvent appear. The structures shown on the <sup>13</sup>C spectra are from manufacturers' literature, from the handbook by Lee and Neville [11], or from the chemical name. We have generally used a standard chemical shorthand for the structures. Some examples follow:

Chemical name	Chemical formula	Brief version
Butoxy group	$\mathrm{CH_3CH_2CH_2CH_2O}$	- \\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\
Glycidyl group	$\mathrm{H_{2}C}^{\mathrm{O}}$ CHC $\mathrm{H_{2}}$ —	
Phenyl ring	$\begin{array}{c} H & H \\ C - C \\ C = C \\ H & H \end{array}$	

Our spectral assignments for most of the <sup>13</sup>C spectra are based on chemical-shift substituent effects obtained from the literature [8,9] or calculated from the spectra of model compounds. We ran the spectra of model compounds if data for them were not available. In a few cases we ran proton-coupled <sup>13</sup>C spectra to aid in the assignments. The line assignments are indicated by a letter near the corresponding carbon atom in the structure. In some cases we have made no assignments either because we do not know the structure or because the spectrum is too complex. No assignments are given for any of the proton spectra.

Appendix A contains carbon-13 NMR line listings of three materials having spectra that are quite complex.

#### **CAUTION**

We remind the user of this catalog of the pitfalls of "fingerprint" spectra comparison. The first is the effect of solvents. In both <sup>13</sup>C and proton spectra, the concentration and the solvent can have widely varying effects on both line shape and position. Another pitfall is that the relative intensities of <sup>13</sup>C spectral lines are notoriously dependent on experimental conditions, such as viscosity and pulse repetition rate. Finally, because most of the materials are standard commercial products, manufacturing impurities, additives, and reaction products formed during storage may be present.

#### EXPERIMENTAL PROCEDURES

Most of the samples were prepared from material on hand. The remainder were either purchased from chemical supply houses or were samples received from manufacturers. The inclusion of a material in this catalog does not imply that the material is still

#### NRL REPORT 8092

made by the manufacturer that we list as the source, that it is only available from that manufacturer, or even that it is still commercially available. Unless otherwise specified, materials were used as received.

Carbon-13 spectra were obtained at 25.15 MHz with a Varian HA-100 spectrometer system, modified for pulsed Fourier transform operation, proton decoupling, and external fluorine-19 field-frequency lock. The pulse interval and number of scans varied with the sample, but were usually 3 to 5 s and 500 scans, respectively. The spectral window was 250 ppm and collection size was either 4000 or 8000 data points. Data were collected and processed with either a Nicolet 1080 or Nicolet 1180 NMR data system. Sample solutions were prepared to obtain maximum concentration consistent with viscosity and solubility considerations. Solvents and approximate solvent concentration by volume are given on each spectrum. Solvent peaks in the spectra are indicated by the symbol x. Chemical shifts are in parts per million from tetramethylsilane (TMS). The solvents, whose chemical shifts relative to TMS were measured separately, served as internal references.

Proton spectra were obtained at 100 MHz on either a Varian HA-100 or Jeol PS-100 spectrometer system. The sweep width was 10 ppm, and spectra were recorded at 0.01 ppm/s. Sample concentrations were 10% or less by volume. The internal reference was TMS.

#### OTHER NMR SPECTRA CATALOGS

Listed here are the proton and <sup>13</sup>C NMR spectra catalogs that we know about. We would appreciate hearing of any which we have not listed.

#### Proton

- 1. Varian Associates High Resolution NMR Spectra Catalog, Combined Vols. 1 and 2, N.S. Bhacca, D.P. Hollis, L.F. Johnson, E.A. Pier, and J.N. Shoolery, Varian Associates, Palo Alto, Calif., 1962 and 1963
- 2. The Sadtler Guide to the NMR Spectra of Polymers, W.W. Simons and M. Zanger, Sadtler Research Laboratories, Inc., Philadelphia, Pa., 1973
- 3. Sadtler Standard Nuclear Magnetic Resonance Spectra, Sadtler Research Laboratories, Inc., Philadelphia, Pa., (continuing)\*
- 4. The Aldrich Library of NMR Spectra, C.J. Pouchert and J.R. Campbell, Aldrich Chemical Company, Inc., Milwaukee, Wis., 1974.
- 5. Catalog of Nuclear Magnetic Resonance Spectral Data (American Petroleum Research Project 44 and Manufacturing Chemists Association Research Project), Chemical Thermodynamic Properties Center, Texas A&M University, College Station, Tex. (Loose leaf data sheets, extant.)

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<sup>\*</sup>Sadtler also markets a number of specialized smaller NMR spectra collections.

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#### Carbon-13

- 1. Carbon-13 NMR Spectra, L.F. Johnson and W.C. Jankowski, John Wiley and Sons, Inc., New York, N.Y., 1972
- 2. Sadtler Standard Carbon-13 NMR Spectra, Sadtler Research Laboratories, Inc., Philadelphia, Pa., 1976

#### REFERENCES

- F.A. Bovey, High Resolution NMR of Macromolecules, Academic Press, Inc., New York, 1972.
- 2. J. Schaefer, Chap. 4, in *Topics in Carbon-13 NMR Spectroscopy*, Vol. 1 (G.C. Levy, editor), Wiley Interscience, New York, 1974.
- 3. J. Schaefer, E.O. Stejskal, and R. Buchdahl, Polym. Prepr. Div. Polym. Chem., 17, No. 2, 17 (1976).
- 4. A.N. Garroway, W.B. Moniz, and H.A. Resing, ACS Coatings and Plastics Preprints 36, No. 2, 133 (1976).
- 5. C.F. Poranski, Jr., and W.B. Moniz, ACS Coatings and Plastics Preprints 36, No. 2, 139 (1976).
- 6. J.A. Pople, W.G. Schneider, and H.J. Bernstein, *High Resolution Nuclear Magnetic Resonance*, McGraw Hill Book Co., Inc., New York, 1959.
- 7. J.W. Emsley, J. Feeney, and L.H. Sutcliffe, *High-Resolution Nuclear Magnetic Resonance Spectroscopy*, Pergamon Press, New York, 2 vols. 1965, 1966.
- 8. J.B. Stothers, Carbon-13 NMR Spectroscopy, Academic Press, New York, 1972.
- 9. G.C. Levy and G.L. Nelson, Carbon-13 Nuclear Magnetic Resonance for Organic Chemists, Wiley-Interscience, New York, 1972.
- 10. T.C. Farrar and E.D. Becker, *Pulse and Fourier Transform NMR*, Academic Press, Inc., New York, 1971.
- 11. H. Lee and K. Neville, *Handbook of Epoxy Resins*, McGraw Hill Book Co., Inc., New York, 1967.

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#### Appendix A

#### CARBON-13 NMR LINE LISTINGS FOR THREE MATERIALS

In this we give the complete line listings for three materials for which the spectra are quite complex because of the presence of several structural isomers. These materials are 3,4-epoxycyclohexylmethyl-(3,4-epoxy)cyclohexane carboxylate (see Spectrum 23), 3,4-epoxy-6-methylcyclohexylmethyl-3,4-epoxy-6-methylcyclohexane carboxylate (see Spectrum 24), and methyl-4-endo-methylene tetrahydrophthalic anhydride (see Spectrum 50). In the case of the anhydride, additional complexity may arise from the presence of dicarboxylic acids formed by hydrolysis. Line positions in parts per million from TMS follow:

1. 3,4-Epoxycyclohexylmethyl-(3,4-epoxy)cyclohexane carboxylate:

~~ =	~~ =	
22.7	29.7	52.4
24.5	31.7	52.7
25.2	33.9	53.1
25.5	37.4	53.3
26.1	39.1	69.5
27.9	51.6	175.3
28.7	51.9	175.8
28.9	52.2	

 $2.\ 3, 4\hbox{-}Epoxy-6\hbox{-}methylcyclohexylmethyl-}3, 4\hbox{-}epoxy-6\hbox{-}methylcyclohexane\ carboxylate:}$ 

17.1	29.8	49.6
18.9	30.9	50.5
19.5	31.2	50.8
23.8	32.3	51.1
24.5	33.2	51.4
25.6	34.3	51.7
26.3	40.0	173.1
26.6	35.2	65.1
27.8	38.3	65.6
28.9	43.2	173.7
29.4	45.9	174.1
	18.9 19.5 23.8 24.5 25.6 26.3 26.6 27.8 28.9	18.9     30.9       19.5     31.2       23.8     32.3       24.5     33.2       25.6     34.3       26.3     40.0       26.6     35.2       27.8     38.3       28.9     43.2

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#### ${\bf 3.\ Methyl-4-} endo{\bf .methylene\ tetrahydrophthalic\ anhydride:}$

15.0	49.2	130.2
16.3	50.0	135.4
43.5	50.4	136.6
46.0	50.6	137.8
46.4	51.0	139.6
46.7	51.4	142.6
46.9	51.5	146.5
47.1	52.7	148.0
47.3	55.2	171.5
48.0	70.4	171.8
48.2	127.0	172.1

#### INDEX OF MATERIALS

The materials in this index are listed in alphabetical order by chemical names or chemical type. If the sample has a particular manufacturer's product designation, it is listed under that designation also. The "see" cross reference refers to a material that has a  $^{13}\mathrm{C}$  spectrum that is virtually identical to the spectrum of the material listed. The "similar to" cross reference refers to a material that has a spectrum that differs only slightly from the spectrum of the material listed.

S <sub>I</sub> Name	pectrum No.	Name S	pectrum No.
2-aminoethanol	31 33 33 43 43	N,N-dimethylbenzylamine N,N-dimethylethylenediamine sym-dimethylethylenediamine 4,8-dioxatricyclo[5.1.0.0 <sup>3,5</sup> ] octane	. 40 . 28 . 28
Araldite 502	9 50 3 17 40	4-DI-PIP	. 37 . 41 . 19
chlorendic anhydride	49 50 12 20 38	Epi-Rez SU-8	. 8 . 2 71.
D.E.N. 431		Epon 1007	. 13 . 24 . 22
diaminodiphenyl sulfone	43 32 17 30 25	4-epoxy)cyclohexyl carboxylate 4-(1,2-epoxyethyl)-1,2- epoxycyclohexane	. 22 hyl-
diglycidyl ether of tetrabromo- bis-phenol A	11 41	carboxylate	. 18

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anavy vasin DCFDA tumor		G M105	_
epoxy resin, DGEBA type: Araldite 7071	3	Genepoxy M195	7
D.E.R. 332. See D.E.R. 332LC.		H-221	34
D.E.R. 332LC	1	norbornene-2,3-dicarboxylic	
D.E.R. 661. Similar to		anhydride	49
Araldite 7071. Epon 828	2	hexahydrophthalic anhydride hexamethylenetetramine	48 35
	4	HHPA	48
Epon 1001. Similar to Araldite 7071.		3,3'-imino-bis-propylamine	29
Epon 1004	4	Kopox 159	16
Epon 1007	5	maleic anhydride	45
epoxy resin, DGEBA type plus		1,4-bis(methylamino)cyclohexane.	38
butyl glycidyl ether;		methyl-4-endo-methylene tetrahydrophthalic anhydride	50
Epon 815	8	4,4'-methylenedianiline	42
di-n-butyl phthalate; Araldite 502	0	MXDA	39
	9	MY720	17
bis-(2,3-epoxycyclopentyl) ether; ERL 2258	C	NMA. Similar to CIBA 906.	
n-octyl glycidyl ether;	6	PAPA	44
Genepoxy M195	7	phthalic anhydride	46
epoxy resin, DGEBF type	12	piperidine	36
epoxy resin, polyfunctional	10	polyazelaic polyanhydride	44
bis(2,3-epoxycyclopentyl)ether,		polyglycidyl ether of tetraphenylene ethane	13
	21	polyglycoldiamine	34
4-(2,3-epoxy)propoxy- <i>N</i> , <i>N-bis</i> (2, 3-epoxypropyl)-aniline	15	resorcinol diglycidyl ether	16
	19	tetraethylenepentamine	27
N,N-bis(2,3-epoxypropyl)-2,4,	1.4	tetraglycidyl methylenedianiline	17
	14 21	tetrahydrophthalic anhydride	47
	15	tridimethylaminomethyl phenol	41
	20	triethylenetetramine	26
ERL-2258	6	triglycidyl-p-aminophenol	15
ERLA-4221	23	4,4'-trimethylene-dipiperidine vinyl cyclohexene dioxide	37 22
ERX-67	14	m-xylenediamine	39
ethanolamine	31	ZZLB 0822	34

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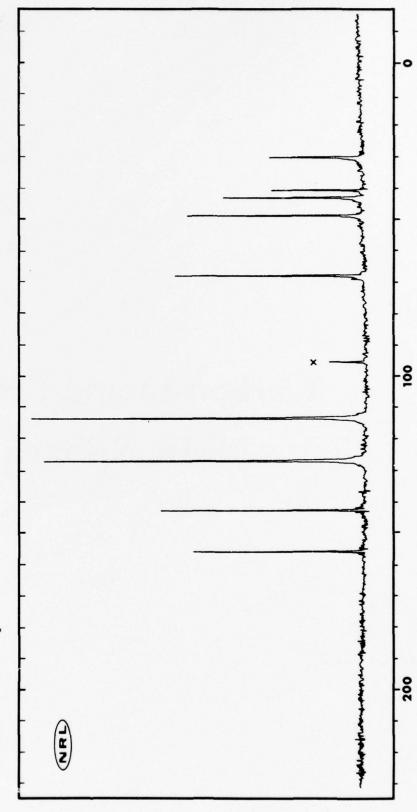
# Carbon-13 and Proton NMR Spectra

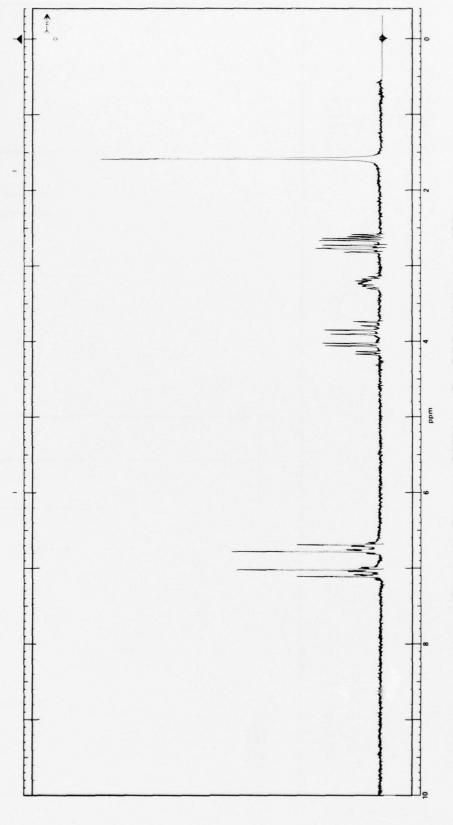
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Epoxy Resin, DGEBA Type

Assignments: a 30.1 h 142.4 b 40.7 i 155.4 c 43.1	e 68.1 f 113.2	g 126.7
	Source: Dow D.E.R. 332LC	Solvent: 50% CC1 <sub>4</sub> ×

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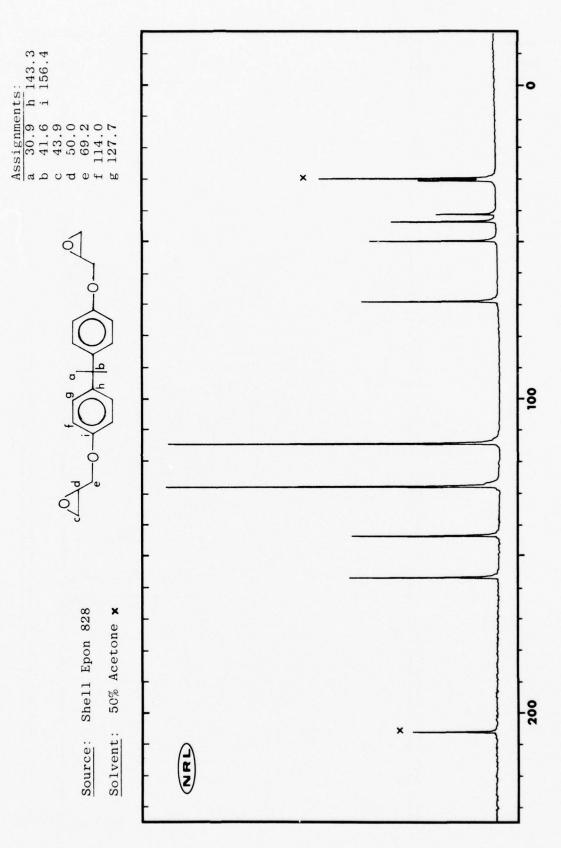


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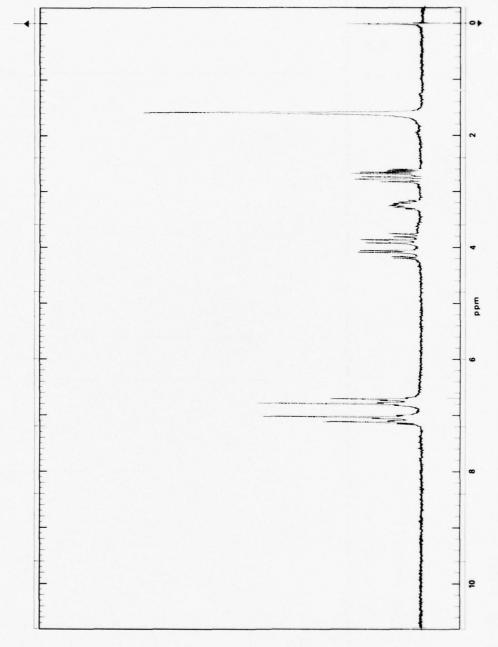
Spectrum 1 — Epoxy resin, DGEBA type (Dow D.E.R. 332LC); solvent:  $\,$  CDC1  $_3$ 

Epoxy Resin, DGEBA Type

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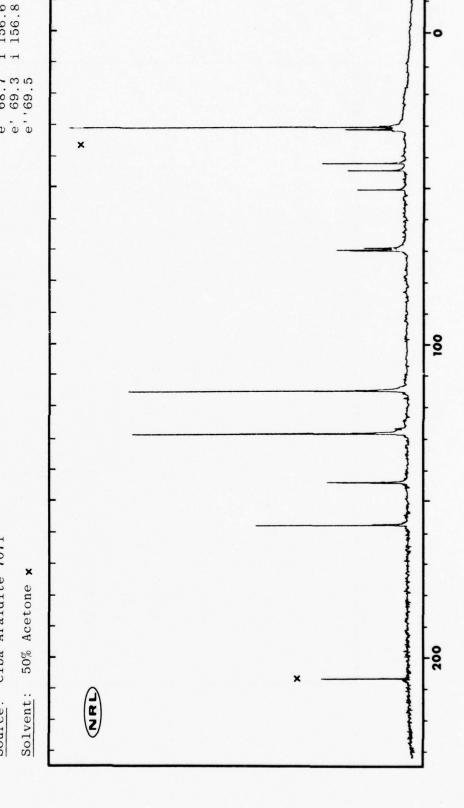


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Spectrum 2 -- Epoxy resin, DGEBA type (Shell Epon 828); solvent: CDC1<sub>3</sub>

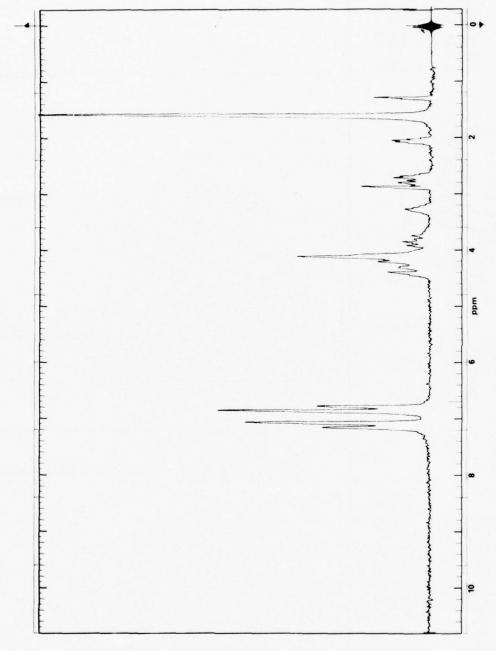
Assignments:
a 30.9 f 114.1
b 41.6 g 127.7
c 44.0 h 143.2
d 50.1 h'143.4
e 68.7 i'156.6
e' 69.3 i 156.8 0 Ciba Araldite 7071 Epoxy Resin, DGEBA Type Source:

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Spectrum 3 — Epoxy resin, DGEBA type (Ciba Araldite 7071); solvent: acetone-d $_6$ 

Epoxy Resin, DGEBA Type

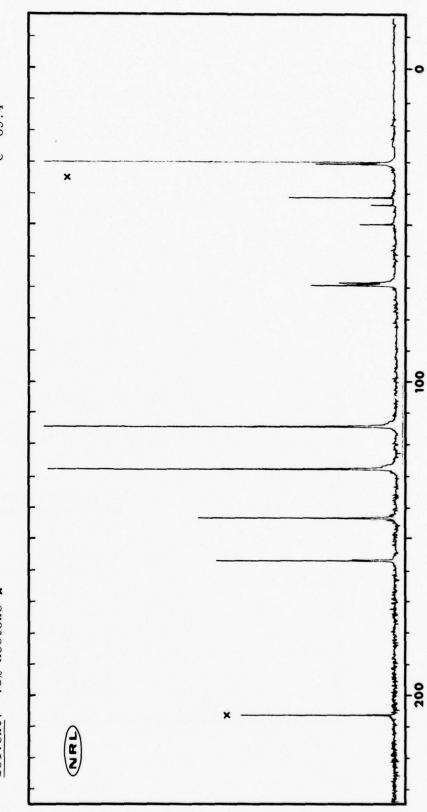
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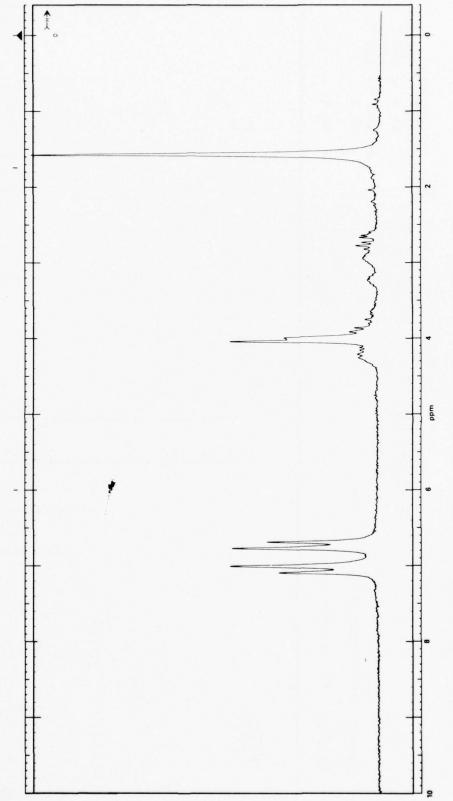
f 114.1 g 127.7 h 143.1 h 143.3 i 156.5 i 156.7

Shell Epon 1004 Source:

75% Acetone x Solvent:

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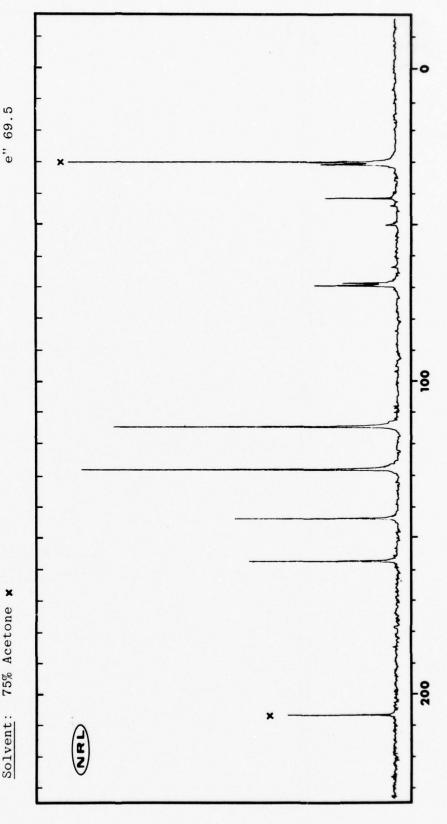


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Spectrum 4 – Epoxy resin, DGEBA type (Shell Epon 1004); solvent:  $\,$  CDC1 $_3$ 

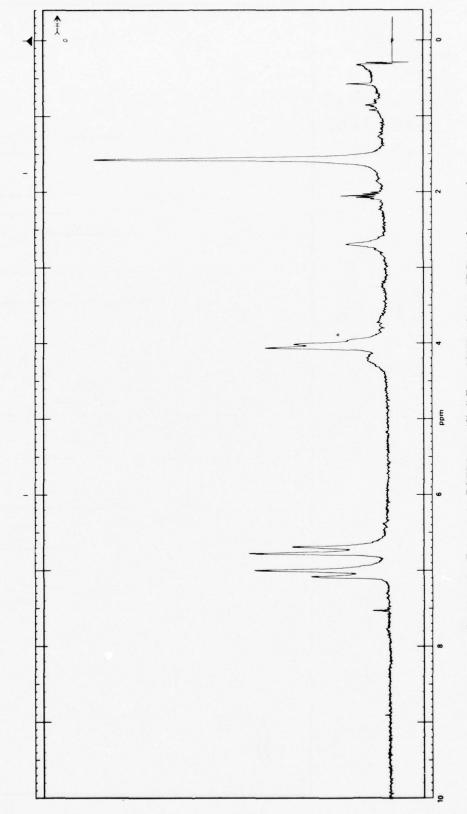
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)	Assignments:	b 41.6 g 128.2	c h 143.2	d 50.0 i 156.7	e 68.7	· •	1 00 11
Epoxy Resin, DGEBA Type	O OHO HO'', I'M		م کے م		Source: Shell Epon 1007		SOJ::0x+: 750 A00+0x0



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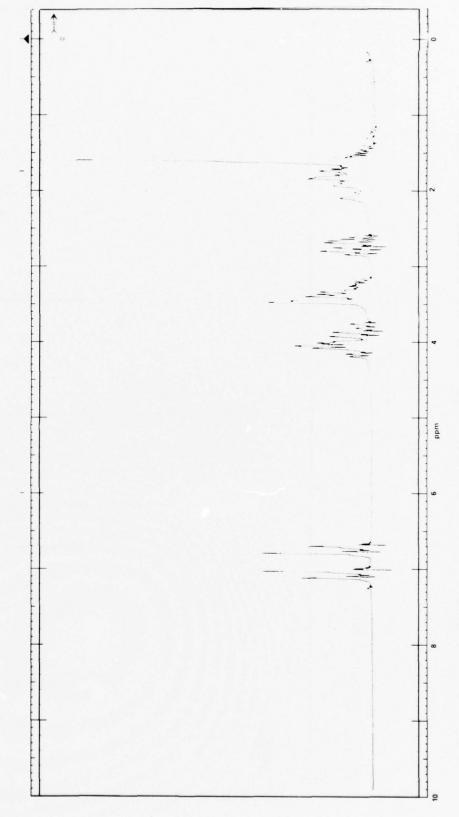
Spectrum 5 - Epoxy resin, DGEBA type (Shell Epon 1007); solvent: CDC13 and acetone-d6

DGEBA Type Epoxy Resin plus Bis(2,3-epoxycyclopentyl)ether

Assignments:  a 24-27.5 b 31.1 i 113.9 c 41.6 j 127.4 d 44.0 k 143.1 e 49.8 l 156.2 f 55-56 g 68.9 h 77-80	The state of the s	-0
10-10-10-10-10-10-10-10-10-10-10-10-10-1	X Marine	001
Source: Union Carbide ERL 2258 Solvent: 25% CCl <sub>4</sub> *	WRD.	200

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Spectrum 6 — DGEBA type epoxy resin plus bis(2,3-epoxycyclopentyl)ether (Union Carbide ERL 2258); solvent: CDC13

DGEBA Type Epoxy Resin plus n-Octyl Glycidyl Ether

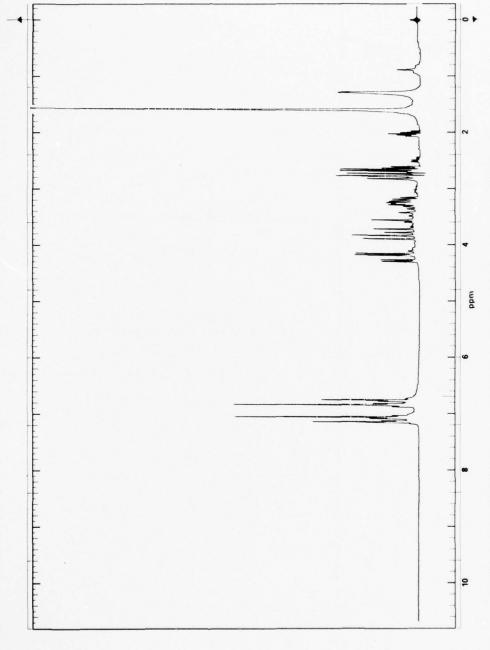
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	3.1						-														
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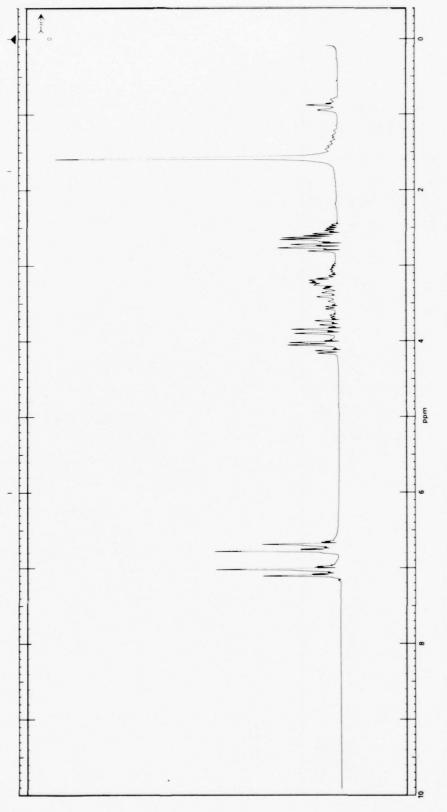
Spectrum 7 — DGEBA type epoxy resin plus n-octyl glycidyl ether (Genepoxy M195); solvent: acetone-d6

DGEBA Type Epoxy Resin plus Butyl Glycidyl Ether

Assignments: a 13.2 h 49.2 o 142.4	d 31.1 k 70.2 e 40 8 1 70.7	E	1.021 11 6.64 8
2 1 0 mm of of of the o	4	) } }	
	n 815	×	m
	Source: Shell Epon	25% CHC1	
	Source:	Solvent	

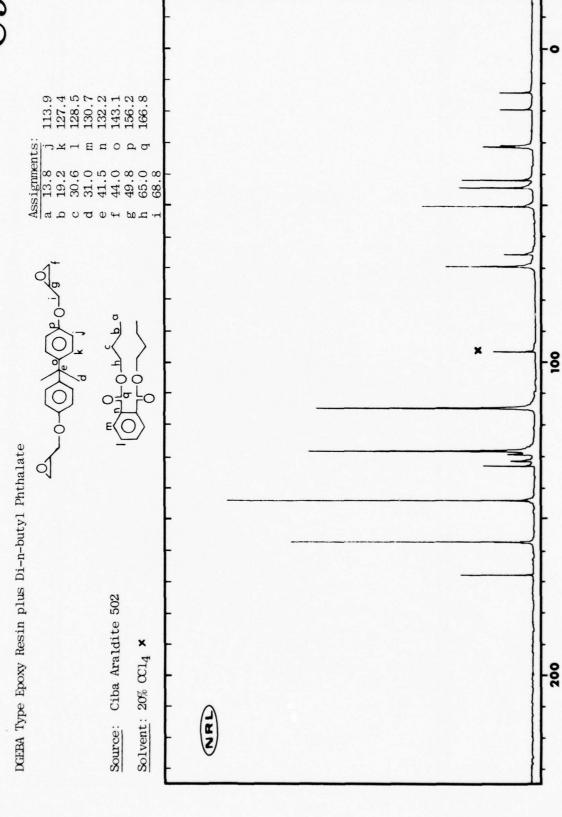
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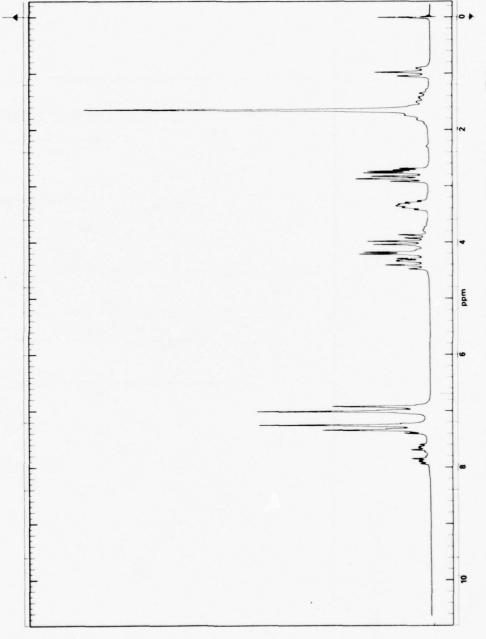


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Spectrum 8 - DGEBA type epoxy resin plus butyl glycidyl ether (Shell Epon 815); solvent: CDC13



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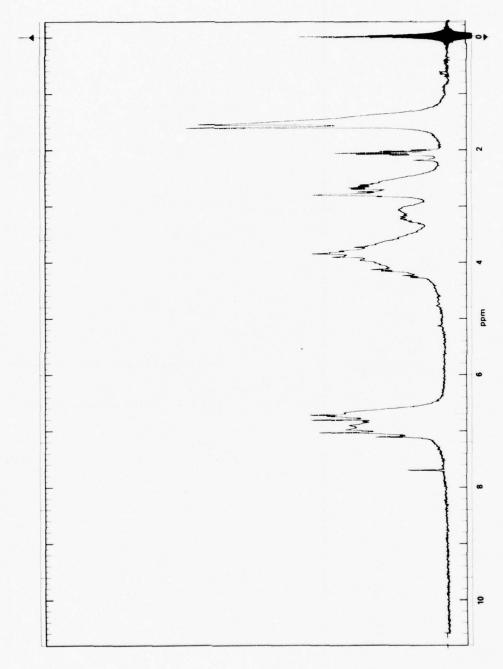
Spectrum 9 - DGEBA type epoxy resin plus di-n-butyl phthalate (Ciba Araldite 502); solvent: CDC13

Epoxy Resin, Polyfunctional

Assignments:  a 30.9 h 124.7 b 41.6 i 127.3 c 43.9 j 128.8 d 50.0 k 134.0 e 69.2 l 142.6 f 111.0 m 154.0 g 113.7 n 156.0	X X	-0
		-
		001
Source: Celanese Epi-Rez SU-8 Solvent: 50% Acetone ×	X XXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXX	200

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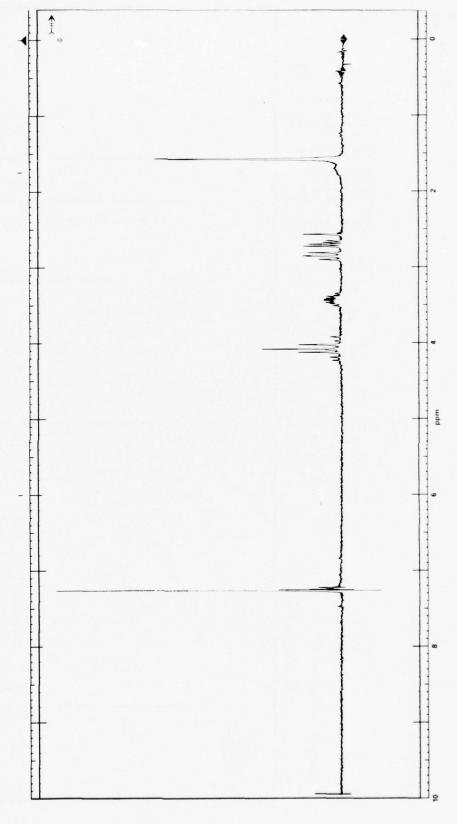
Spectrum 10 - Epoxy resin, polyfunctional (Celanese Epi-Rez SU-8); solvent: acetone-d6 and CDC13 (1:1)

Diglycidyl Ether of Tetrabromobisphenol A

Assignments: a 30.0 h 147.3 b 41.8 i 150.4 c 44.2 d 49.6 e 73.8 f 117.5 g 130.4	- - -		- <b>o</b>
Br Br	- - - -	×	
Source: Dow D.E.R. 542 Solvent: 50% CHCl <sub>3</sub> ×	- (JRN)		200

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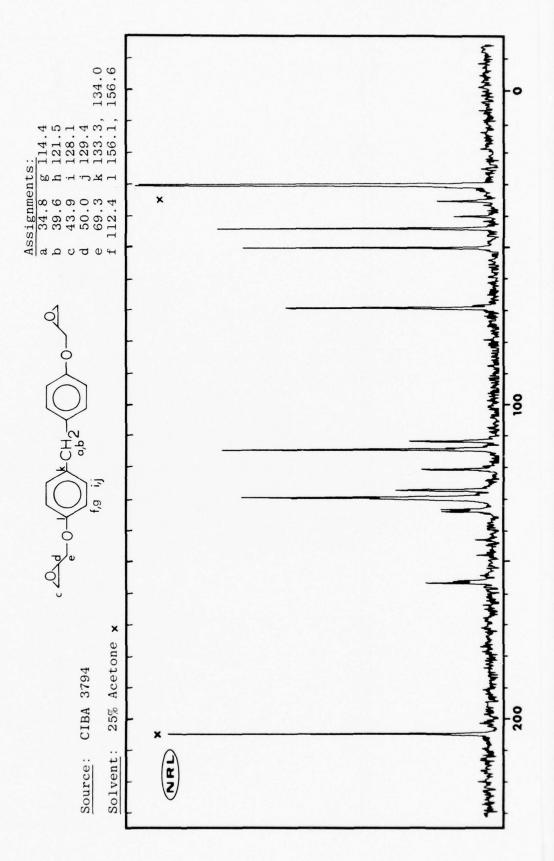
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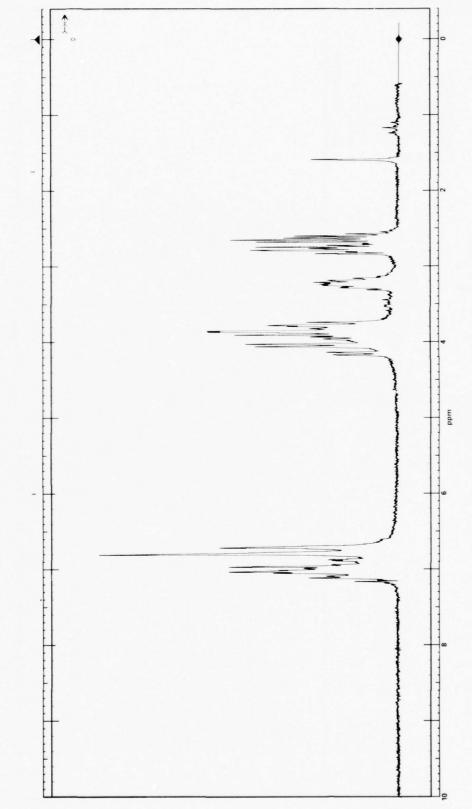
Spectrum 11 — Diglycidyl ether of tetrabromo-bis-phenol A (D.E.R. 542); solvent:  $\,$  CDCl  $_3$ 

Epoxy Resin, DGEBF Type

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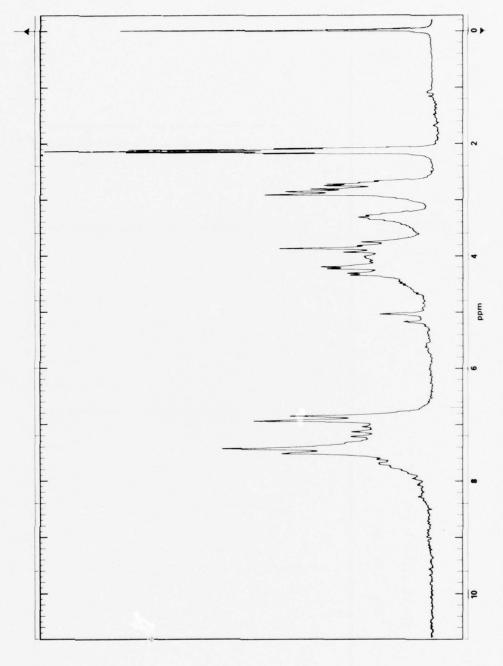
Spectrum 12 — Epoxy resin, DGEBF type (Ciba 3794); solvent: CDC13

Polyglycidyl Ether of Tetraphenylene Ethane

Assignments:  a 44.2 b 49.8 c 68.2 d 113.9 e 128.9 f 136.2 g 155.9 h not observed	•
A-0-0-1-0-0-0-0-0-0-0-0-0-0-0-0-0-0-0-0-	100
Source: Shell Epon 1031 Solvent: 75% CHCl <sub>3</sub> ×	200

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 $Spectrum\ 13-Polyglycidyl\ ether\ of\ tetraphenylene\ ethane\ (Shell\ Epon\ 1031)\ solvent:\ acetone-de$ 

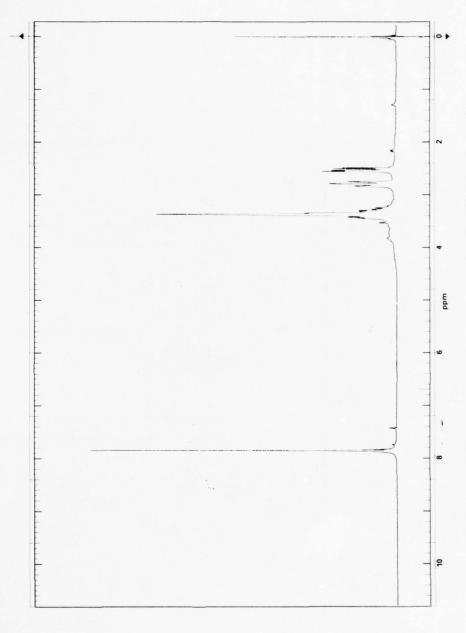
N, N-bis(2, 3-epoxypropyl)-2,4,6-tribromoaniline

Source: Shell ERX-67 Solvent: 50% CHCl <sub>3</sub> x  Solvent: 50% CHCl <sub>3</sub> x   **A 4.7 © 126.9  **Br 44.7 © 126.9  **				
No of the state of	58.1gnments 44.7 e 50.8 g 56.0 56.5 119.3			まっていていたかいのからなるないないのかいかいかいからないないからく
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Solvent: 50% CHCl3 ×  Solvent: 50% CHCl3 ×  WRL	O, R	-		المرابع المراجعة المراجعة
Solvent: 50% CHCl3 x				مي المعويوار المعاملة المعاورة
Solve	e: Shell ERX-67			والمرياب والمراسطة والمراجع وا
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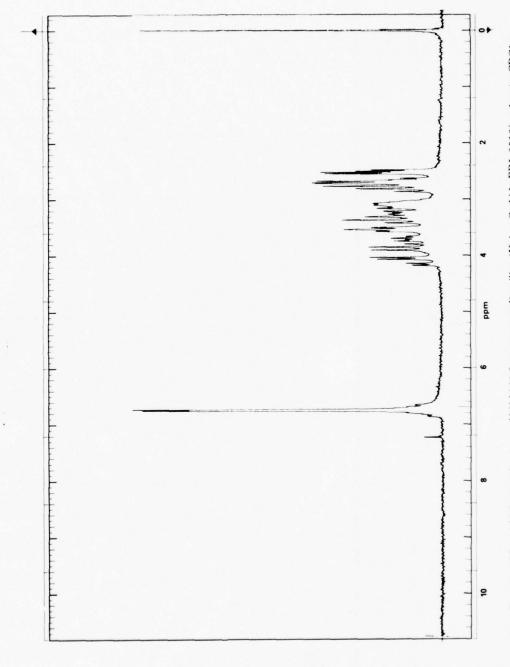
 $Spectrum\ 14-N, N-bis(2,3-epoxypropyl)\cdot 2,4,6-tribromoaniline\ (Shell\ ERX-67); solvent:\ CDC1_3$ 

4-(2,3-epoxy)propoxy-N,N-bis(2,3-epoxypropyl)-aniline

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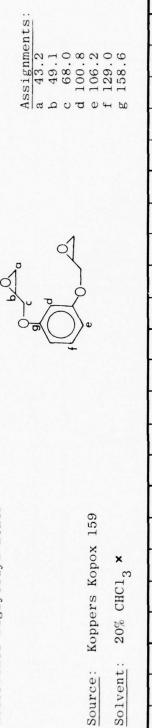


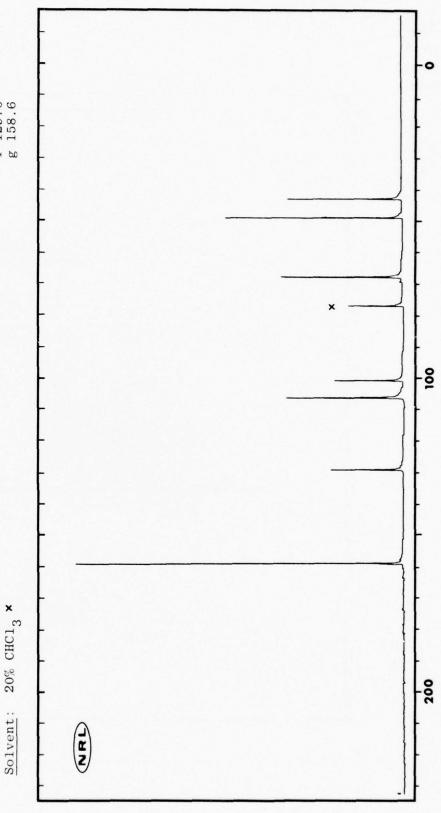
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Spectrum 15 — 4-(2,3-epoxy)propoxy-N,N-bis(2,3-epoxypropyl)-aniline (Union Carbide ERL 0510); solvent: CDC13

Resorcinol Diglycidyl Ether

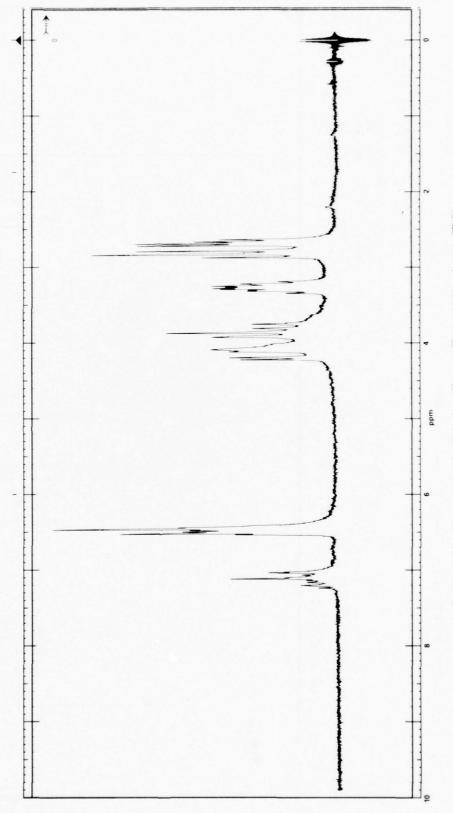
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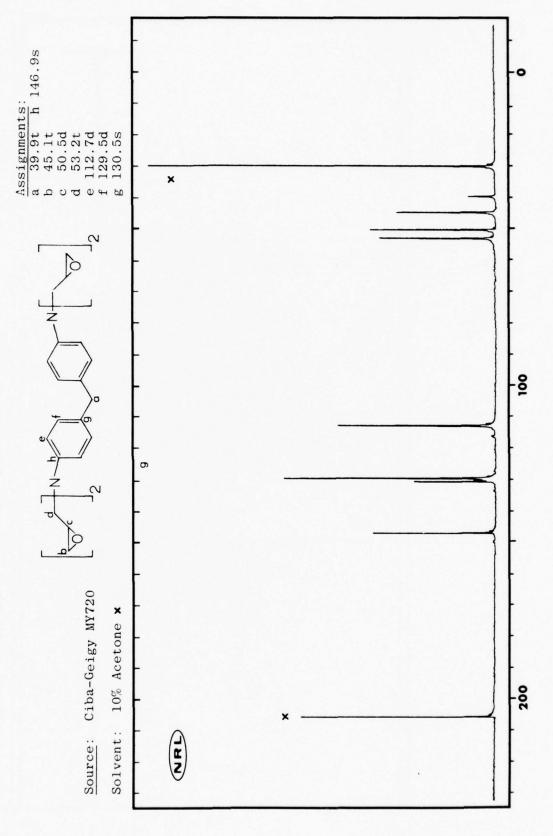


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 $Spectrum\ 16-Resorcinol\ diglycidyl\ ether\ (Koppers\ Kopox\ 159), solvent:\ CDC1_3$ 

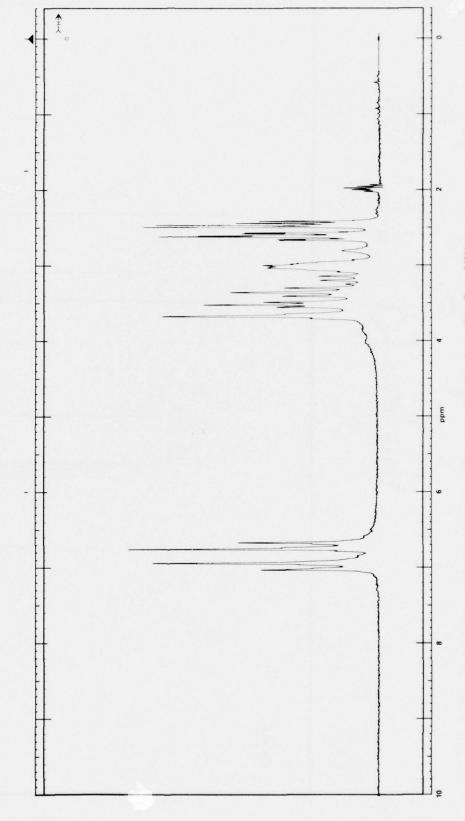
Bis(N,N-di(2,3-epoxypropy1)-4-aminopheny1)methane

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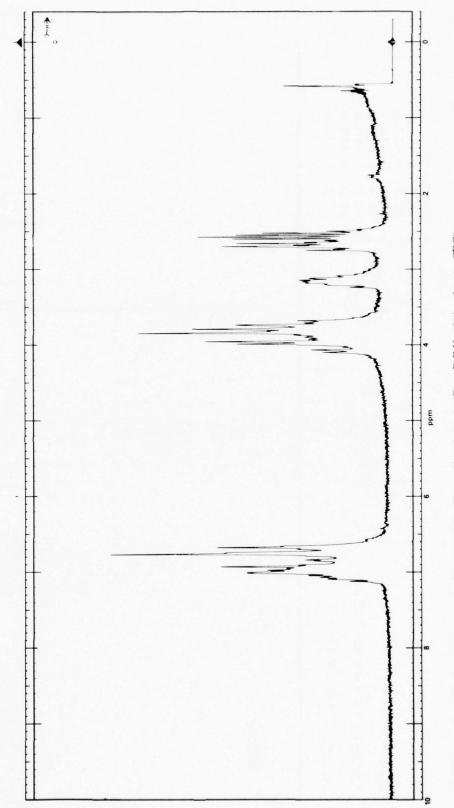
 $Spectrum\ 17-bis(N,N-di(2,3-epoxypropyl)-4-aminophenyl) methane\ (Ciba-Geigy\ MY720); solvent:\ acetone-degree and the contraction of the contra$ 

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Resin				
Epoxy Novolac Resin				
Epoxy				

Source: Dow D.E.N. 431  Solvent: 20% CHCl <sub>3</sub> x  Solvent: 20% CHCl <sub>3</sub> x  NAP.   Solvent: 20% CHCl <sub>3</sub> x  Solvent: 20% CHCl <sub>3</sub> x  NAP.   NAP.  NAP.  Solvent: 20% CHCl <sub>3</sub> x  13.6
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31 31 31 31 31 31 31 31 31 31
COURCE: Dow D.E.N. 431 Colvent: 20% CHCl <sub>3</sub> x

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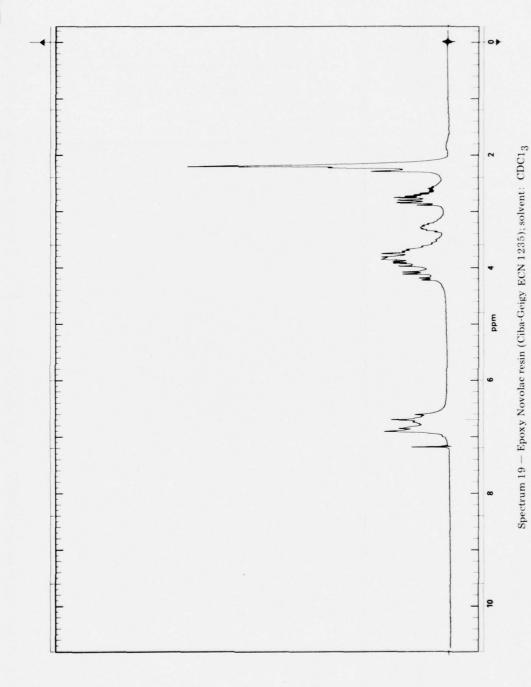
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Spectrum 18 — Epoxy Novolac resin (Dow D.E.N. 431); solvent: CDC13

Epoxy Novolac Resin    Passignments:   Passign			
Ciba-Geigy ECN 1235  So% CHCl <sub>3</sub> ×  So% CHCl <sub>3</sub> ×	10.6 o 152 23.3 p 154 26.0 28.6 30.5 33.1 36.4	and have a second	-0
Ciba-Geigy ECN 1235  So% CHCl <sub>3</sub> ×  So% CHCl <sub>3</sub> ×  So% CHCl <sub>3</sub> ×  So% CHCl <sub>3</sub> ×  Sow CHCl <sub>3</sub> ×	1		
Ciba-Geigy ECN 1235  50% CHCl <sub>3</sub> ×  200	1	X - X	100
clba-Geigy ECN 50% CHCl3 ×	α-0-		-
	rolac Resin Ciba-Geigy ECN 50% CHCl <sub>3</sub> ×	WRD.	200

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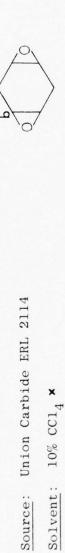
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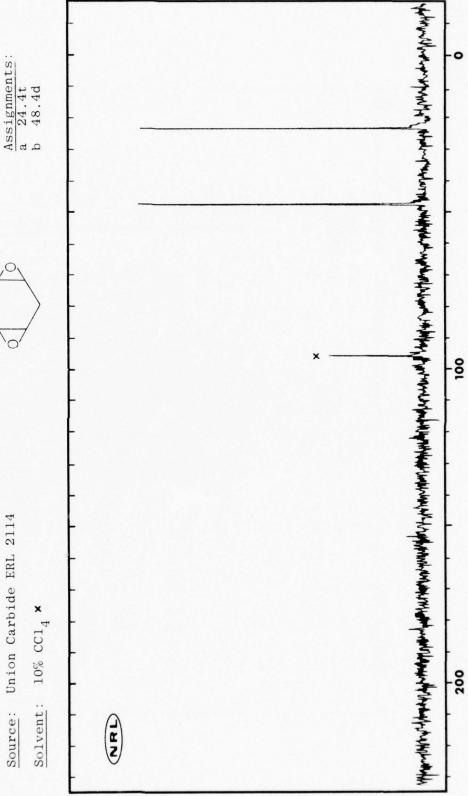
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4,8-Dioxatricyclo  $\begin{bmatrix} 5.1.0.0^3, 5 \end{bmatrix}$  octane

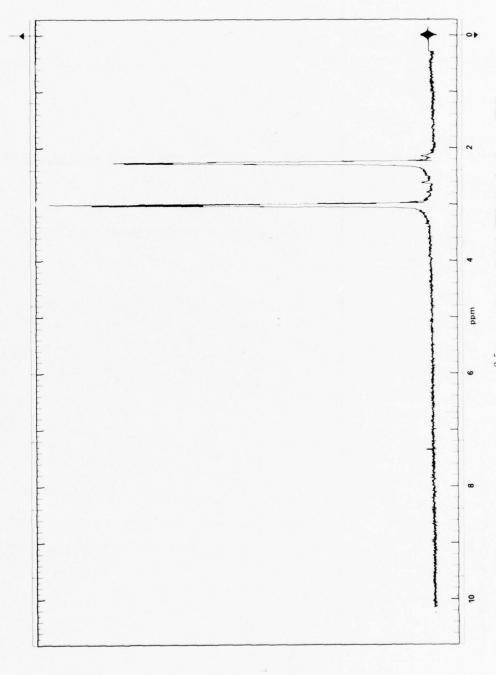
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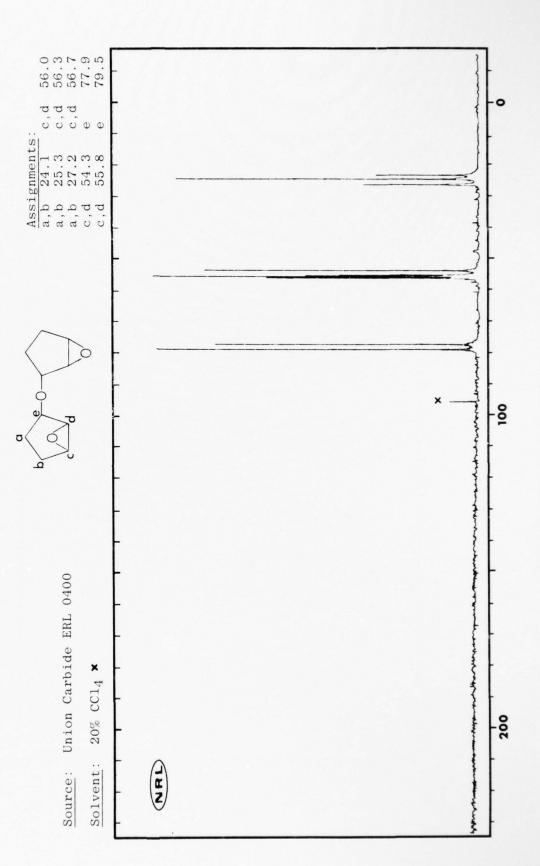


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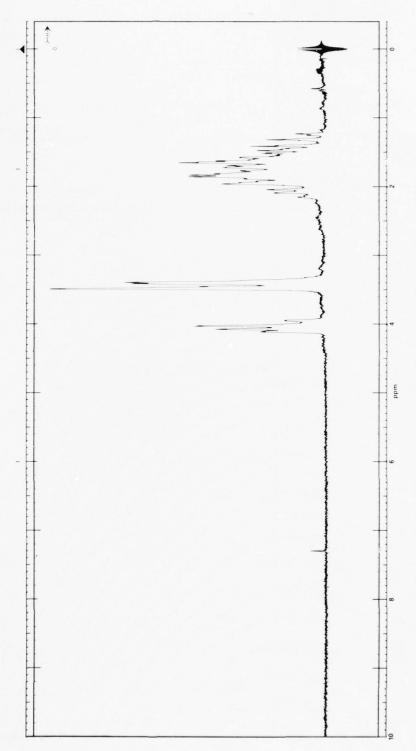
Spectrum 20 --4,8-Dioxatricyclo [5.1.0.03,5] octane (Union Carbide ERL 2114); solvent: CDC13

Bis(2,3-epoxycyclopentyl)ether

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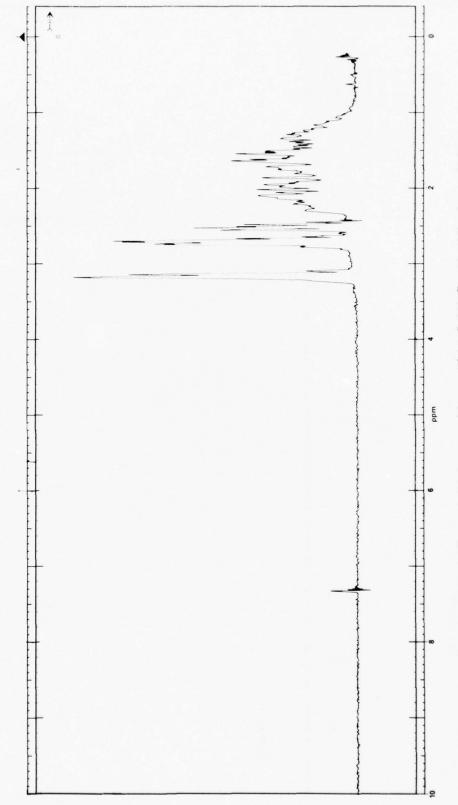
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Spectrum 21 — bis(2,3-epoxycyclopentyl)ether (Union Carbide ERL 0400); solvent: CDC13

34.6 43.6 44.3 49.3 49.9 50.7

Assignments:  a 19.4 h 25.0 o 34.6 b 20.3 i 25.8 p 43.6 c 21.8 j 26.0 q 44.3 d 22.2 k 26.7 r 49.3 e 22.4 l 31.0 s 49.9 f 23.3 m 31.5 t 50.7 g 23.5 n 34.0 u 54.0	The state of the s	0
epoxycyclohexane  Cost State 206	X X The first of t	100
4-(1,2-Epoxyethyl)-1,2-epoxycyc Source: Union Carbide Epoxide Solvent: 25% CHCl <sub>3</sub> *	Į.	200

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 $Spectrum\ 22-4 \cdot (1,2 \cdot Epoxyethyl) \cdot 1,2 \cdot epoxyeyclohexane\ (Union\ Carbide\ Epoxide\ 206); solvent:\ CDC1_3_1, and an expectrum\ CDC1_3_2, and an expectrum\ CDC1_3_3_4, and an expectrum\ CDC1_3_4, and an expectrum\ CDC1_3_5, and an expectrum\ CDC1_5, an$ 

3,4-Epoxycyclohexylmethyl-(3,4-epoxy)cyclohexane Carboxylate

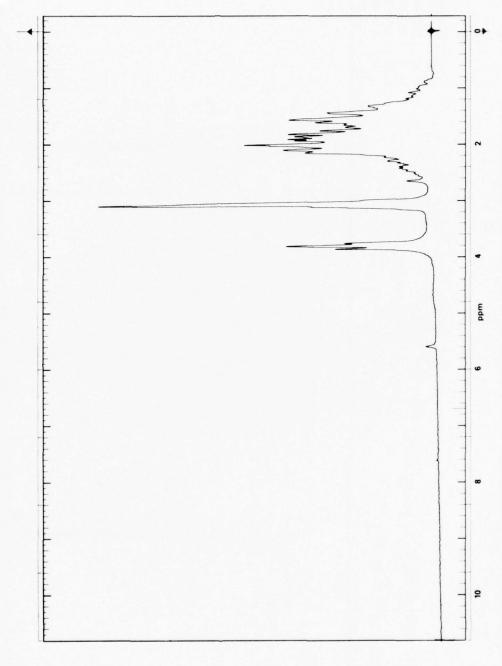
Assignments:  a, a	×	
Source: Union Carbide ERLA 4221		*

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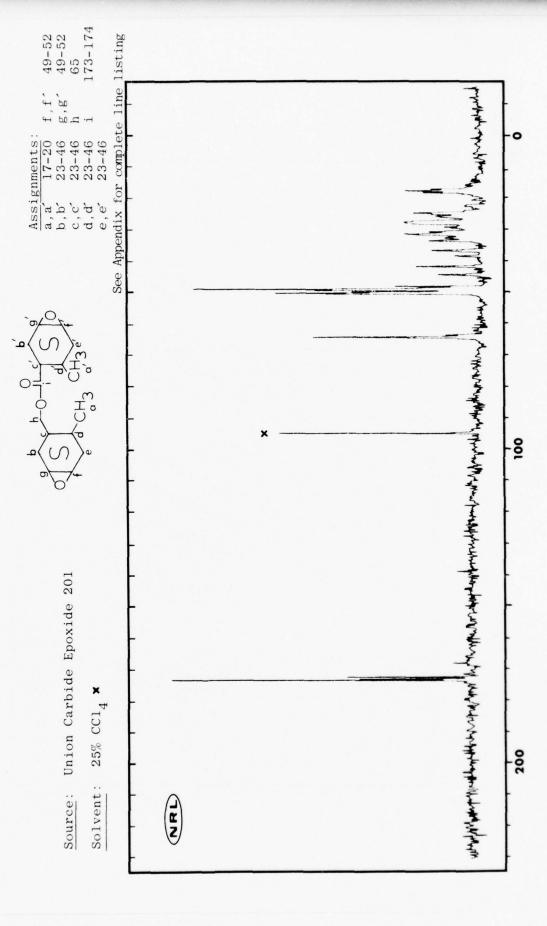


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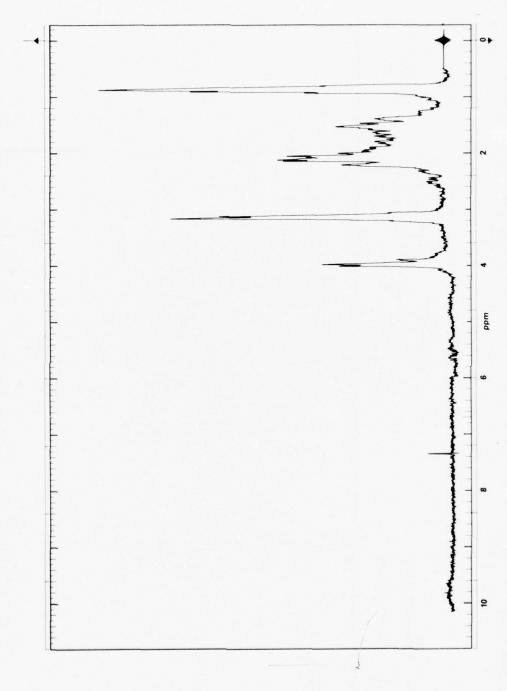
Spectrum 23 — 3,4-Epoxycyclohexylmethyl-(3,4-epoxy)cyclohexane carboxylate (Union Carbide ERLA 4221); solvent: CDC13

3,4-Epoxy-6-methylcyclohexylmethyl-3,4-epoxy-6-methylcyclohexane Carboxylate

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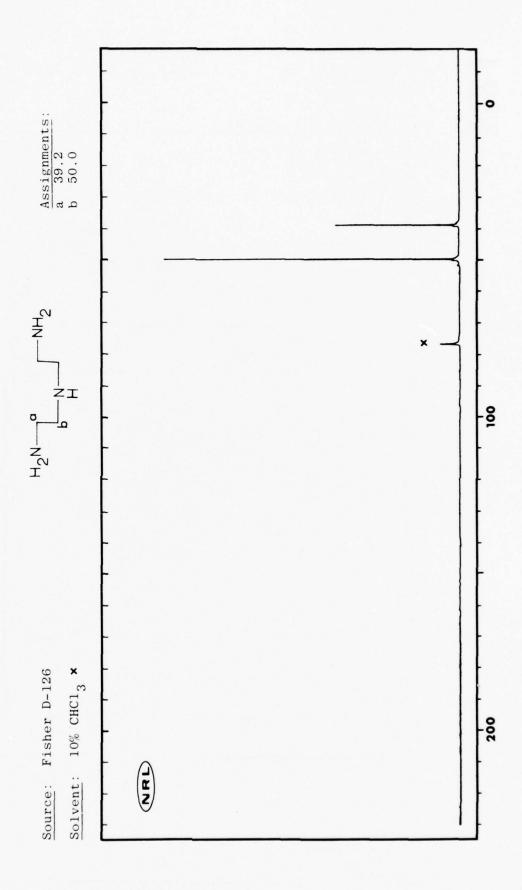
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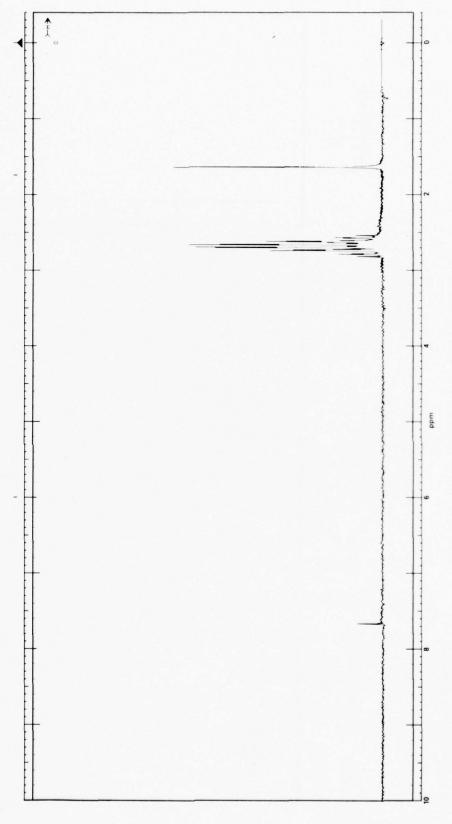
Spectrum 24 - 3,4-Epoxy-6-methylcyclohexylmethyl-3,4-epoxy-6-methylcyclohexane carboxylate (Union Carbide Epoxide 201); solvent: CDC13

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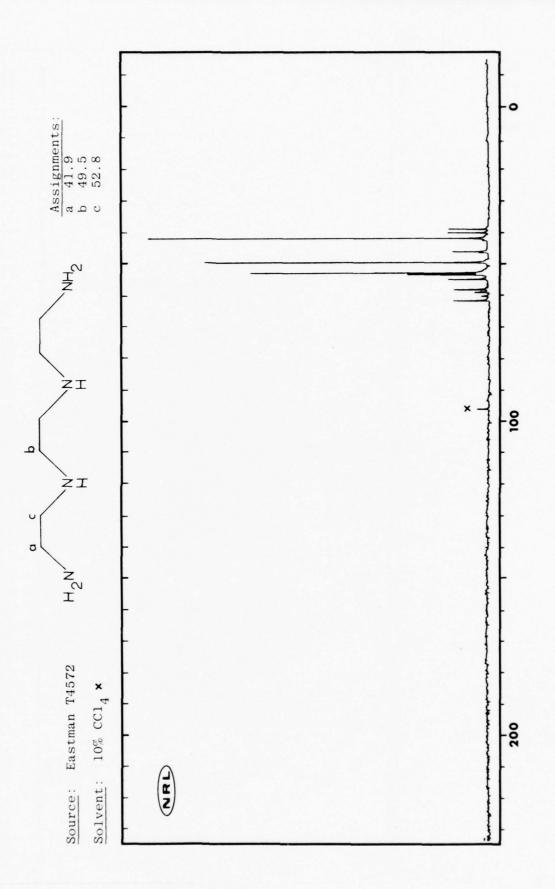


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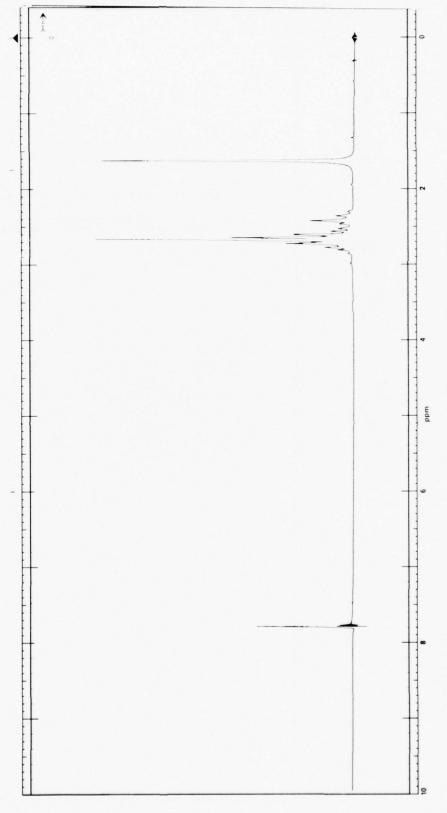
Spectrum 25 - Diethylenetriamine (Fisher D-126); solvent: CDC13

Triethylenetetramine

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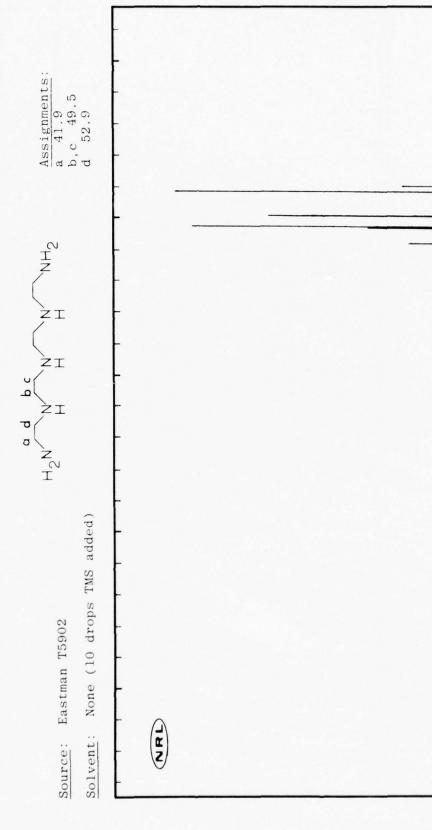


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Spectrum 26 — Triethylenetetramine (Eastman T4572); solvent: CDC13

Tetraethylenepentamine

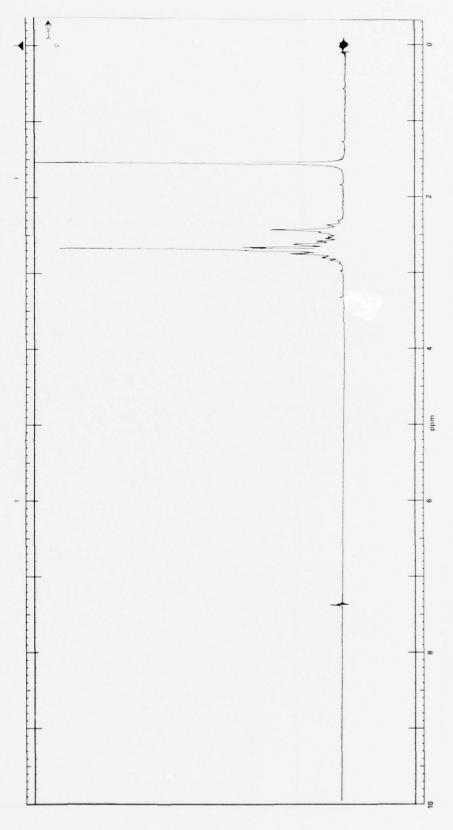
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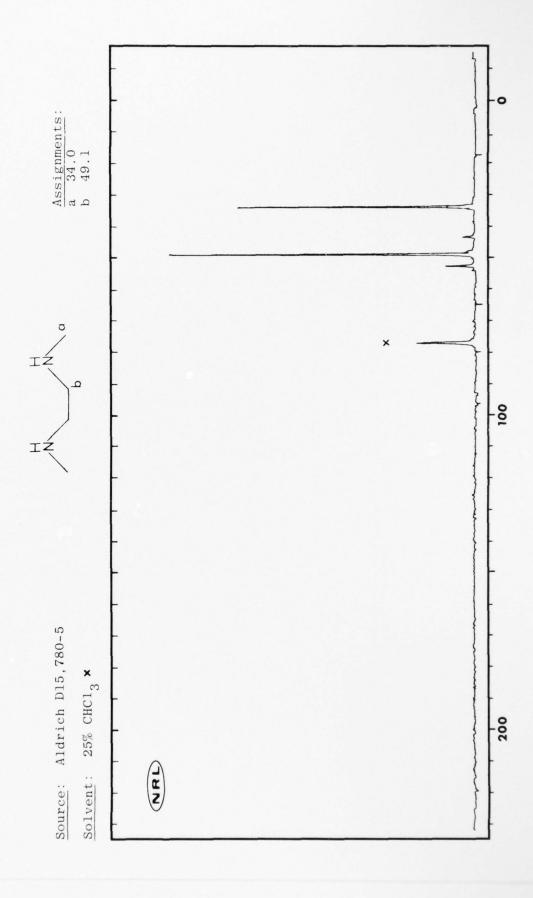


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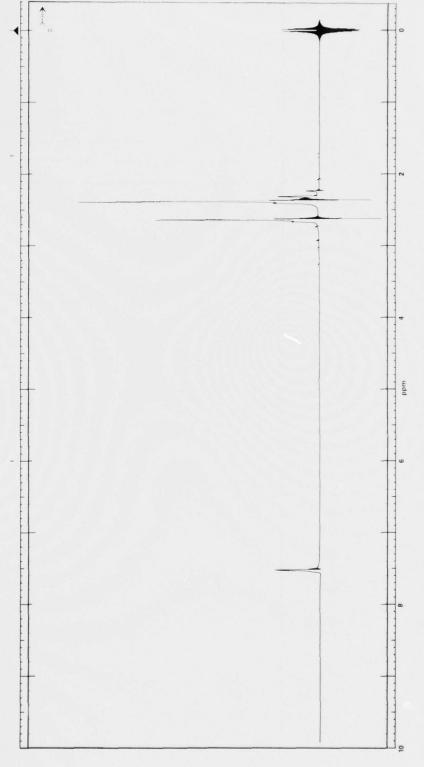
 ${\tt Spectrum~27-Tetraethylenepentamine~(Eastman~T5902); solvent:~CDC1}_3$ 

sym - Dimethylethylenediamine

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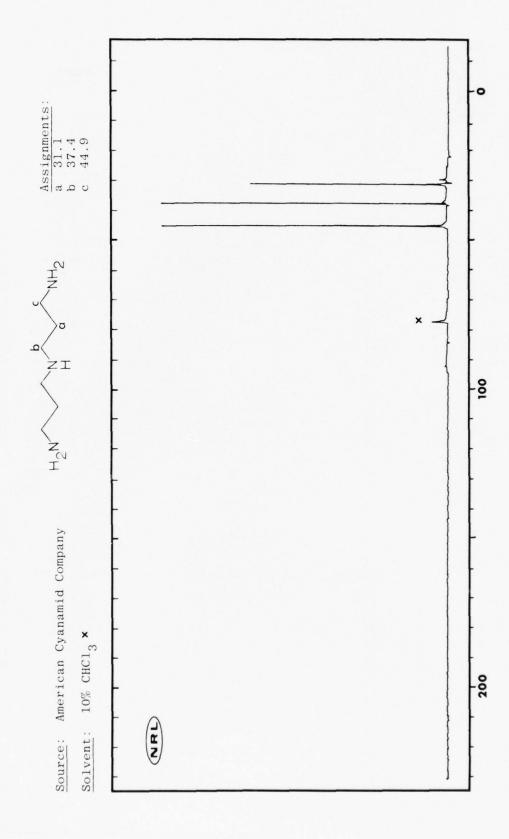


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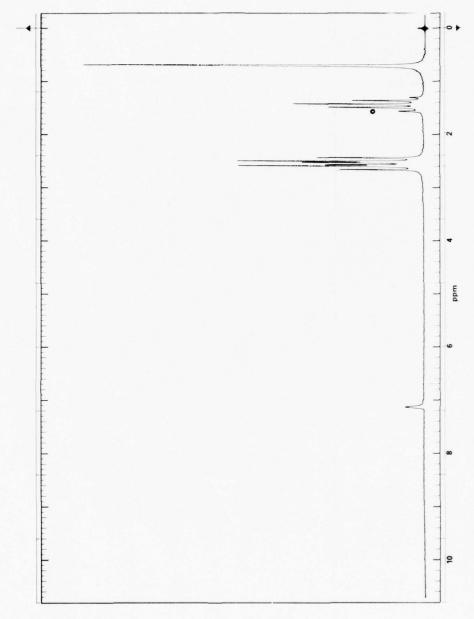


 $Spectrum\ 28-sym\text{-}Dimethylethylenediamine\ (Aldrich\ D15,780-5); solvent:\ CDC1_3$ 

3,3'-Iminobispropylamine

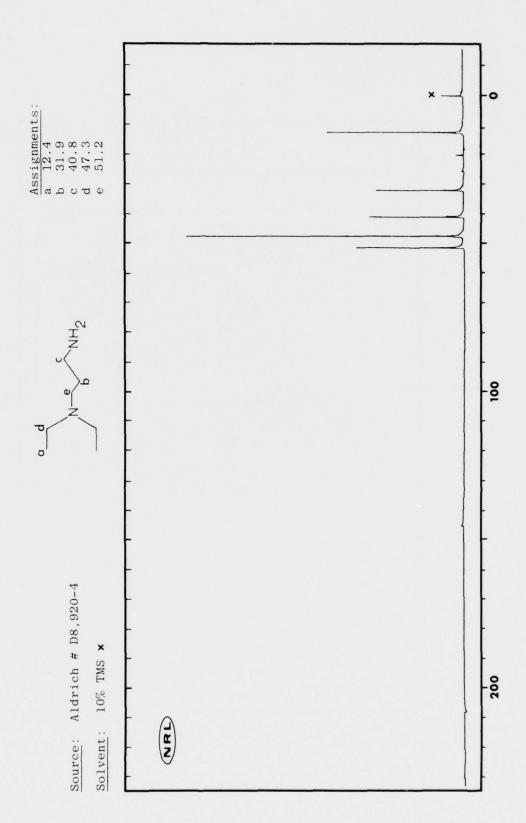


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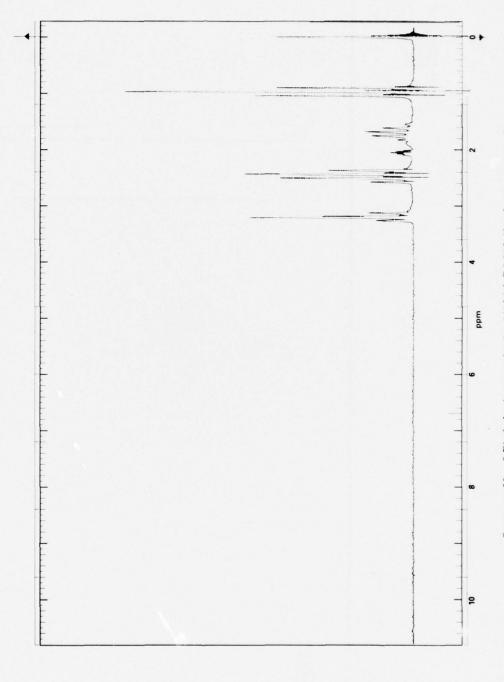


Spectrum 29-3,3'-Imino-bis-propylamine (American Cyanamid Company); solvent: benzene-d6

3-Diethylaminopropylamine



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 $Spectrum \ 30-3- Diethylaminopropylamine \ (Aldrich \ D8,920-4); solvent: \ acetone-d6$ 

2-Aminoethanol

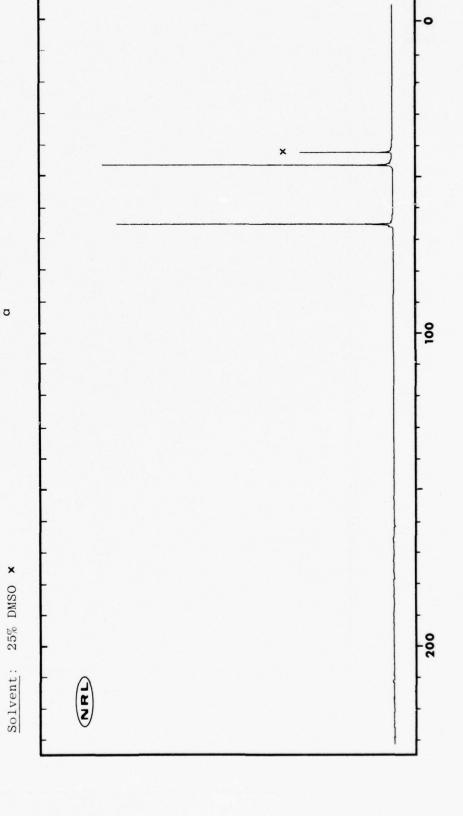
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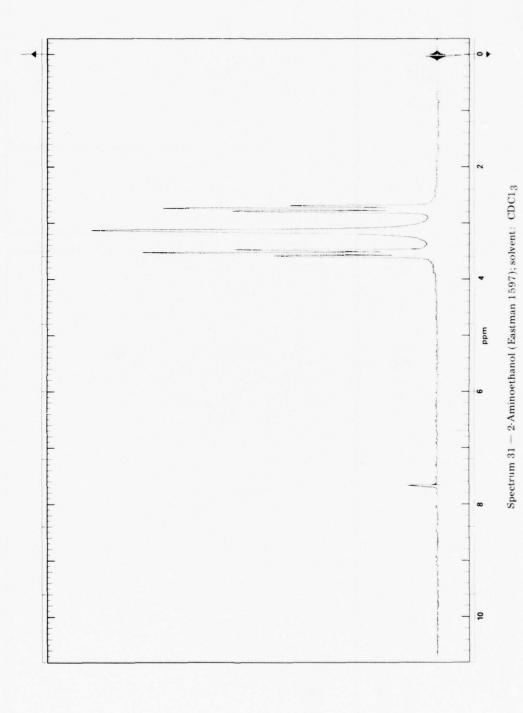
Source: Eastman 1597

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Assignments: a 45.9 b 64.7

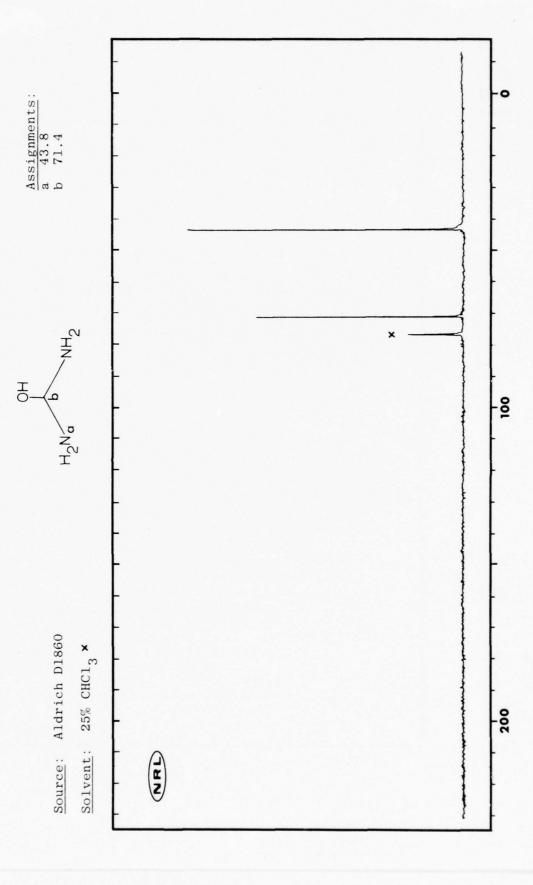


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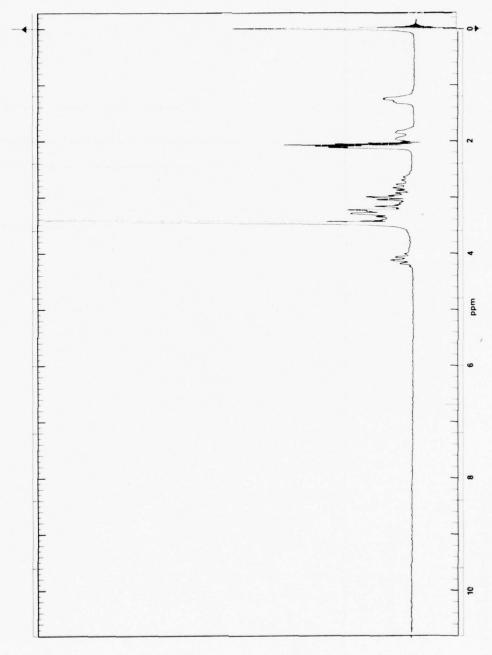
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1,3-Diamino-2-propanol



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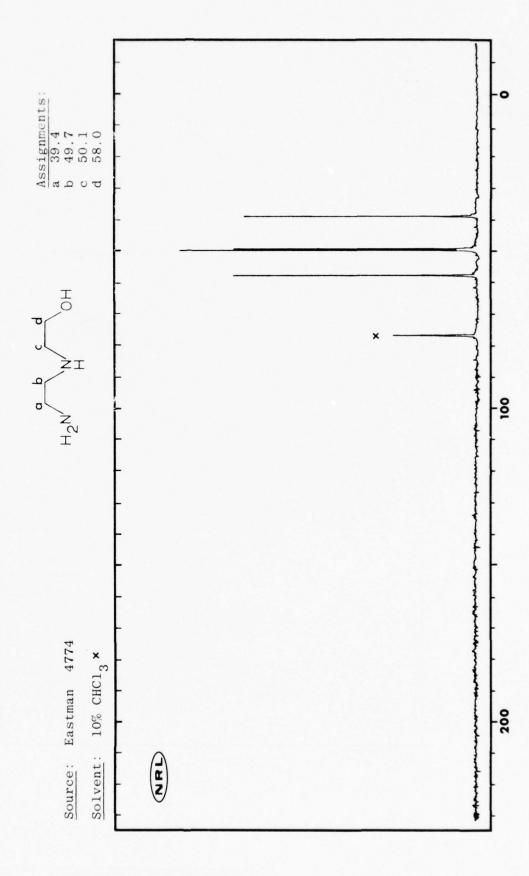


make my make walk to be and the same

 $Spectrum\ 32-1, 3-Diamino-2-propanol\ (Aldrich\ D1860);\ solvent:\ acetone-dg$ 

2-(2-Aminoethylamino)ethanol

The last or world war become in the last of the and



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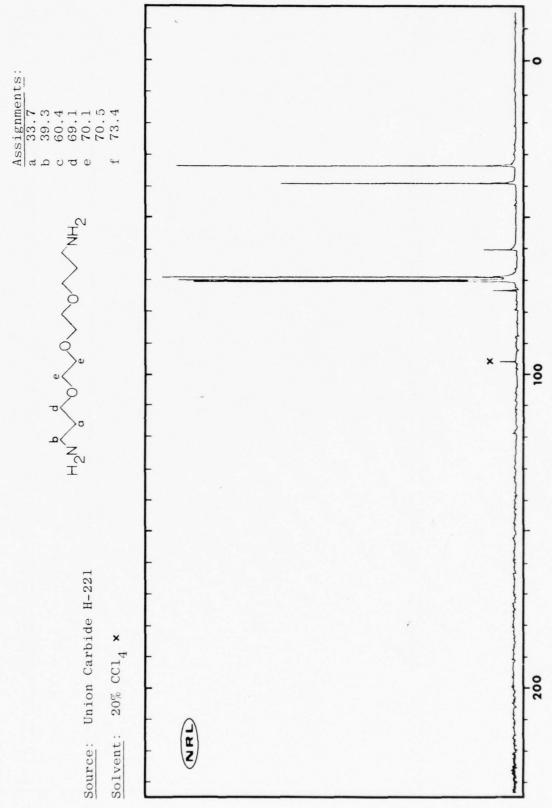


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 ${\tt Spectrum~33-2\cdot(2\cdot Aminoethylamino)ethanol~(Eastman~4774); solvent:~CDC13}$ 

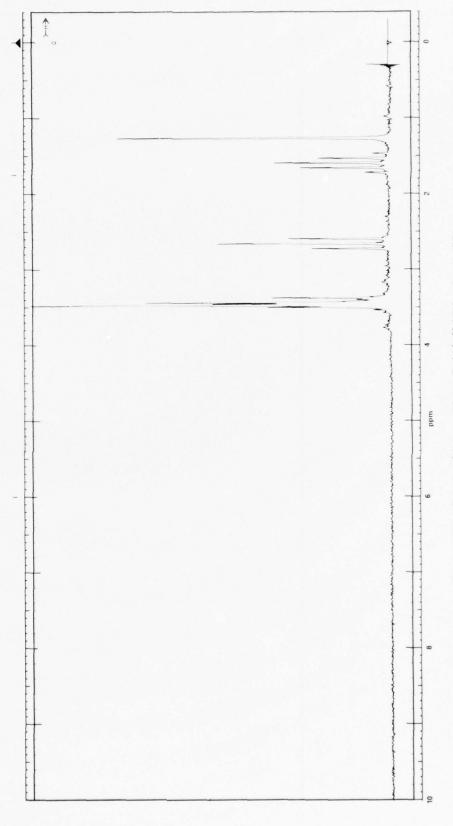
Polyglycol Diamine

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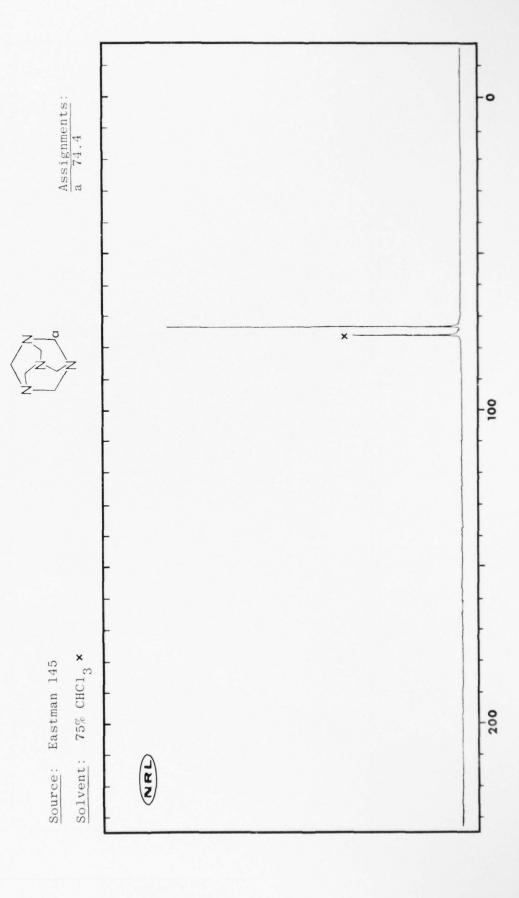


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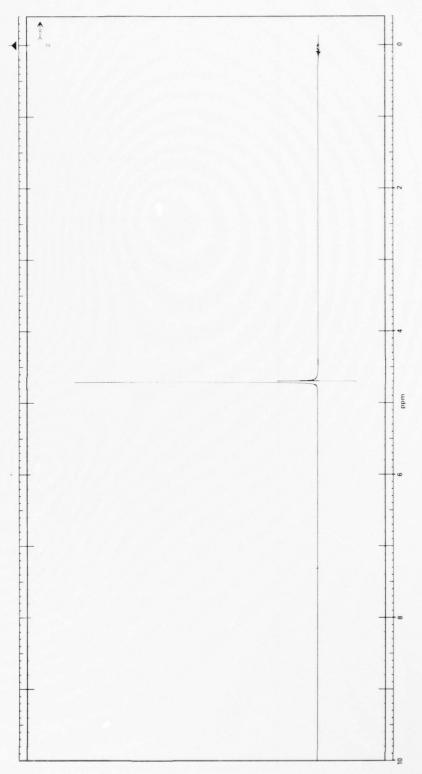
 ${\it Spectrum~34-Polyglycoldiamine~(Union~Carbide~H-221); solvent:~CC1_4}$ 

Hexamethylenetetramine

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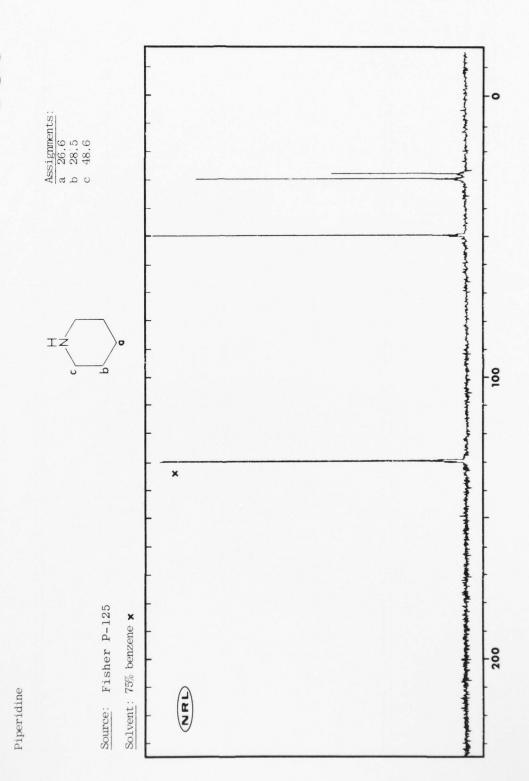


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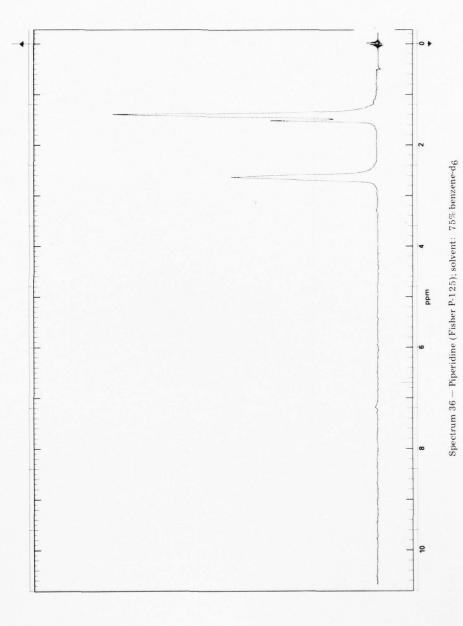
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 $Spectrum\ 35-Hexamethylenetetramine\ (Eastman\ 145); solvent:\ CDC1_{3}$ 



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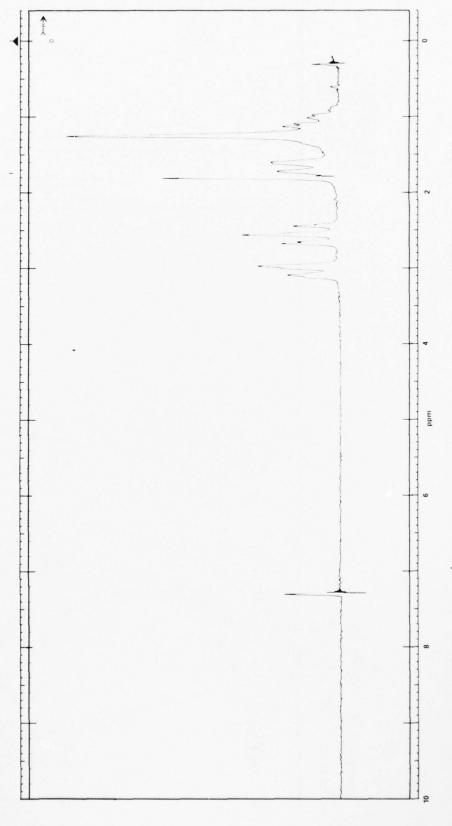
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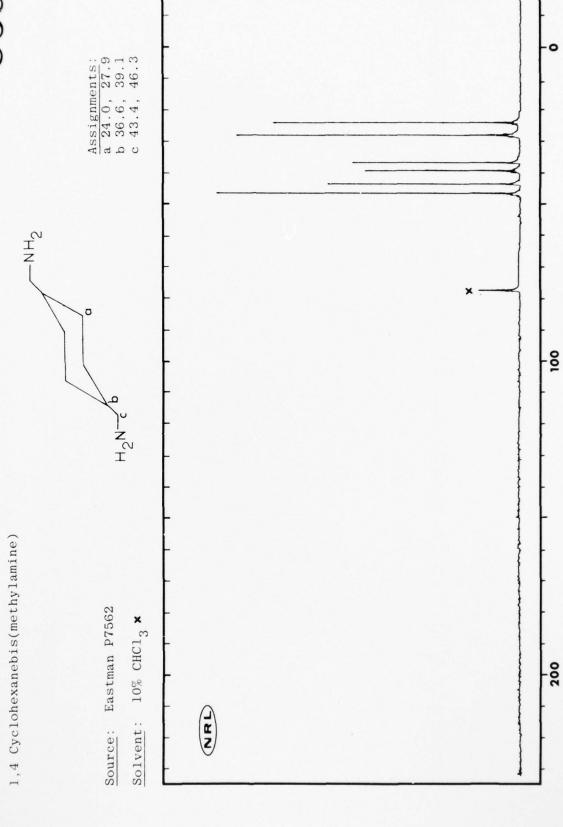
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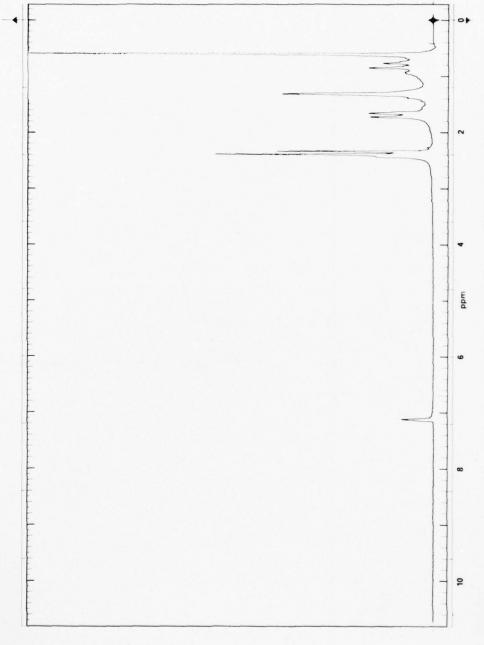
Spectrum 37 - 4,4'-Trimethylene-dipiperidine (Reilly Tar & Chemical Corp. "4-DI-PIP"); solvent: CDC13

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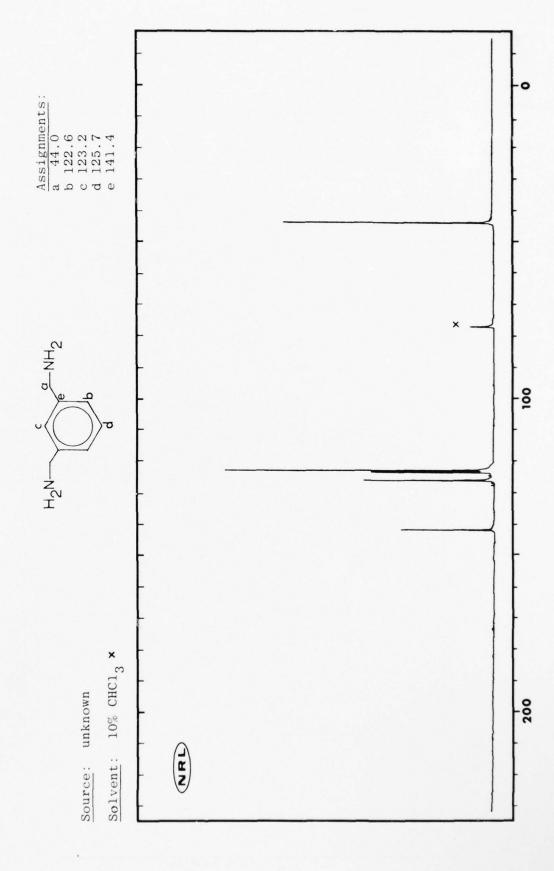
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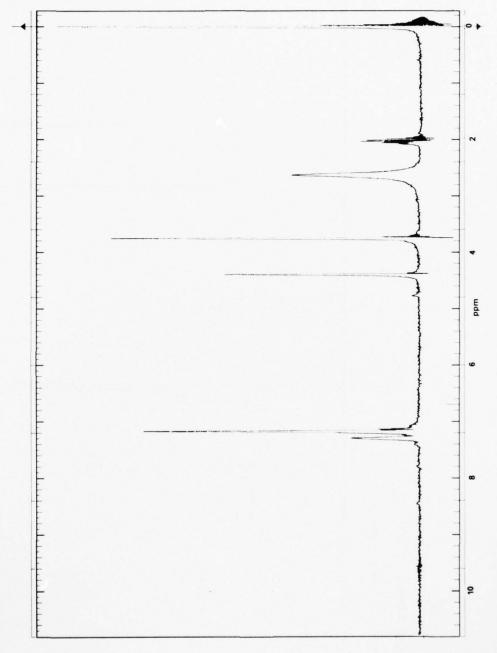
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Spectrum 38-1,4-Cyclohexane-bis(methylamine) (Eastman P7562); solvent: benzene-d6



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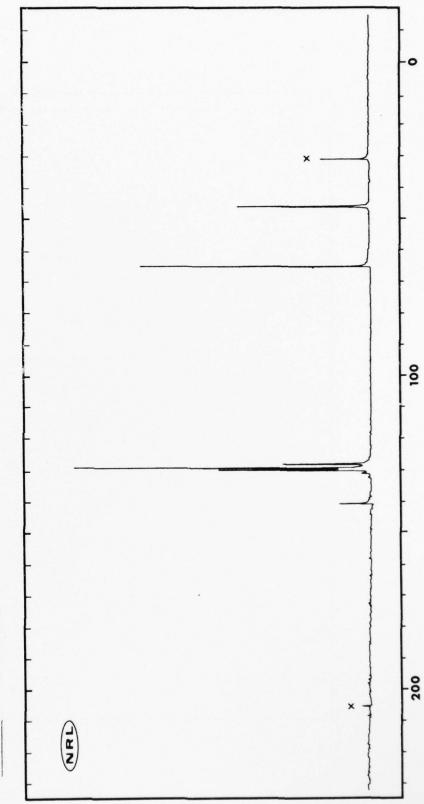
 ${\bf Spectrum~39-m\text{-}Xylenediamine~(source~unknown); solvent:~acetone\text{-}de}$ 

N, N-Dimethyl benzylamıne

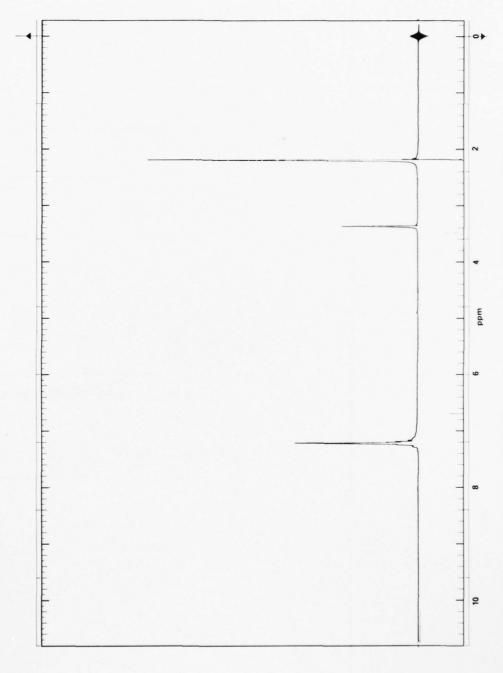
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Spectrum  $40-\textit{N,N-} \textsc{Dimethylbenzylamine} \ (Eastman 1793); solvent: CDCl_3$ 

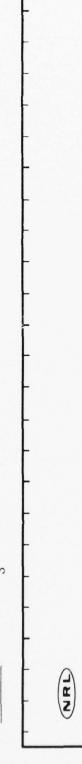
2,4,6-Tris(dimethylaminomethyl)phenol

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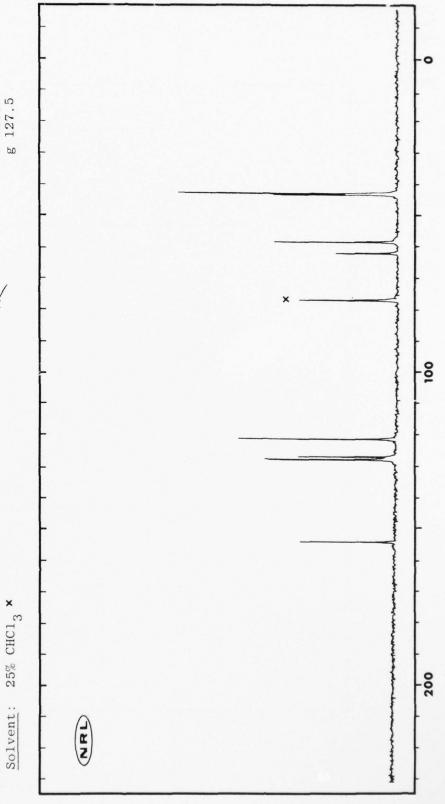
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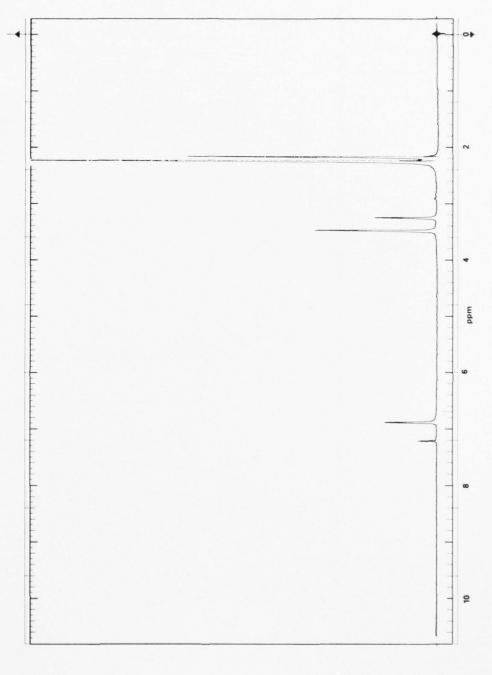
Source: Rohm and Haas DMP-30

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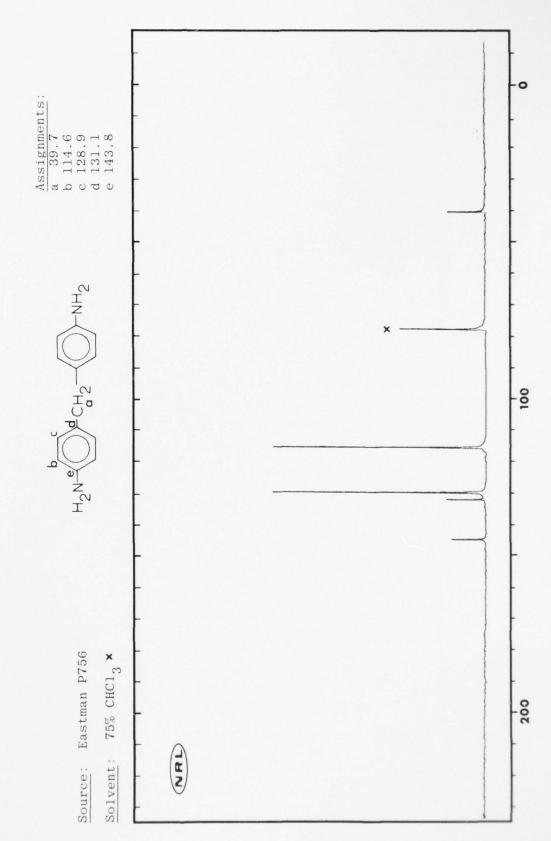


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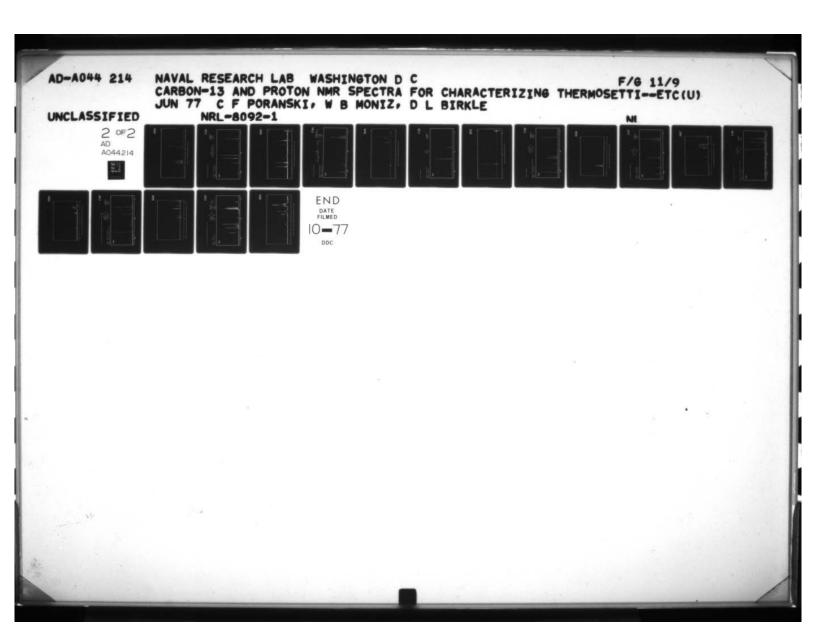
 $Spectrum\ 41-2,4,6-tris(dimethylaminomethyl)\ phenol\ (Rohm\ and\ Haas\ DMP-30);\ solvent:\ CDC1_3$ 

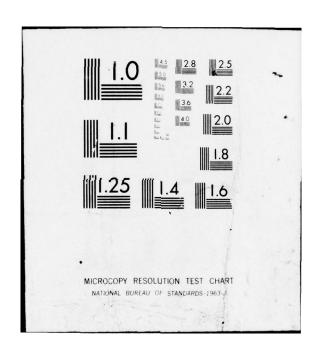
4,4'-Methylenedianiline

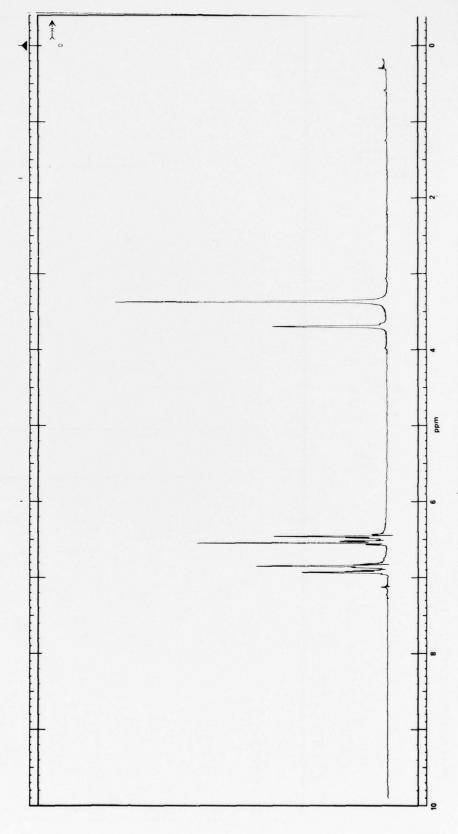
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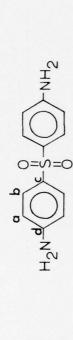


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Spectrum 42-4,4'-Methylenedianiline (Eastman P756); solvent: CDC13

Bis(4-aminophenyl)sulfone

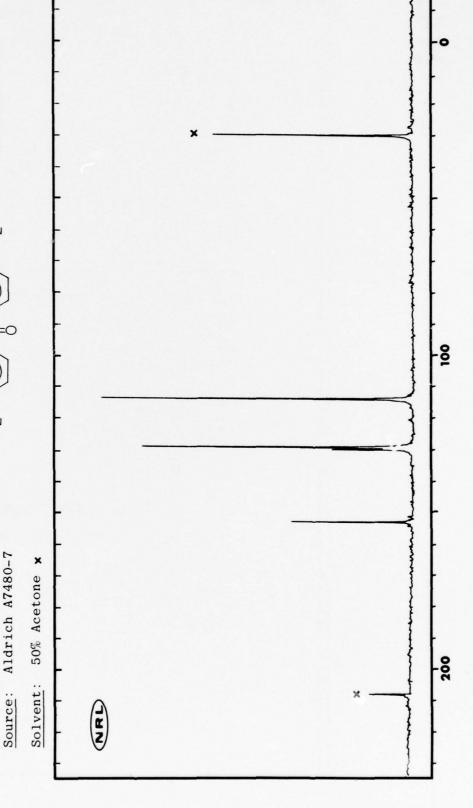
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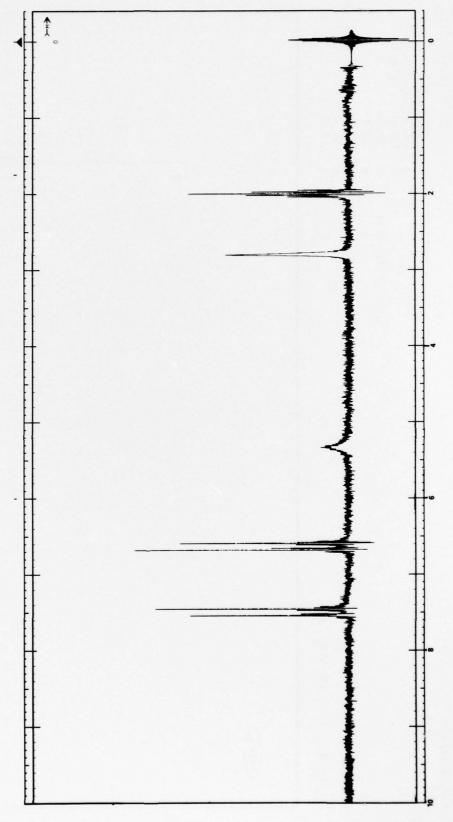


Aldrich A7480-7

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Assignments: a 113.6d b 128.9d c 129.7s d 152.3s



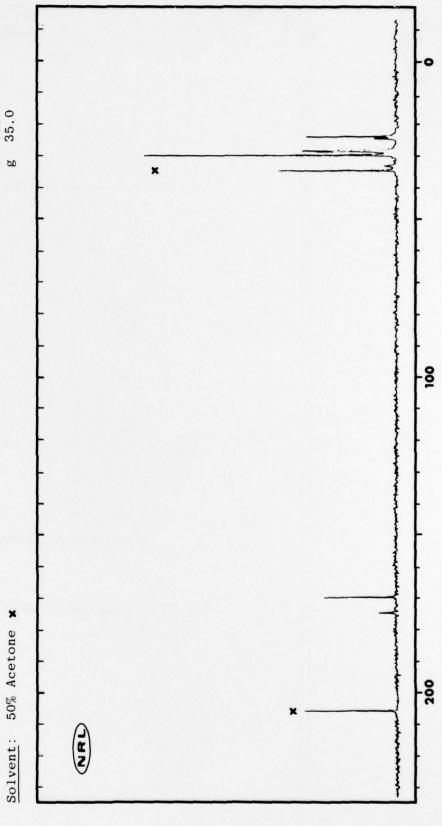


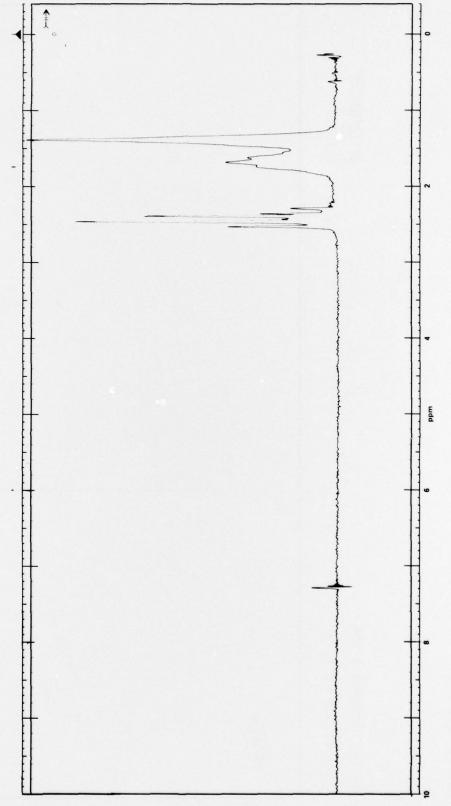
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Spectrum 43-bis(4-aminophenyl)sulfone (Aldrich A7480-7); solvent: acetone-d6

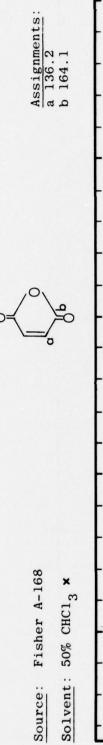
Polyazelaic Polyanhydride

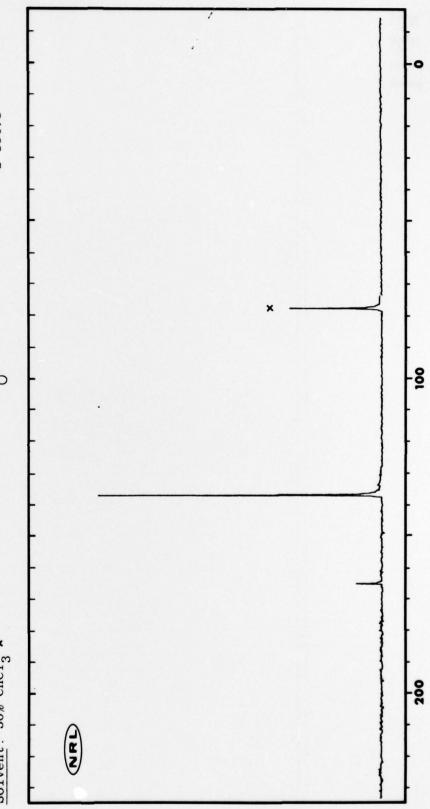
Assignments a 24.4 h b 25.0 i c 28.7 d 29.0	e 29.7 f 33.7	g 35.0	
T	μ		
S OH	Emery 9872 PAPA	Solvent: 50% Acetone x	
	Source: Emery	Solvent:	

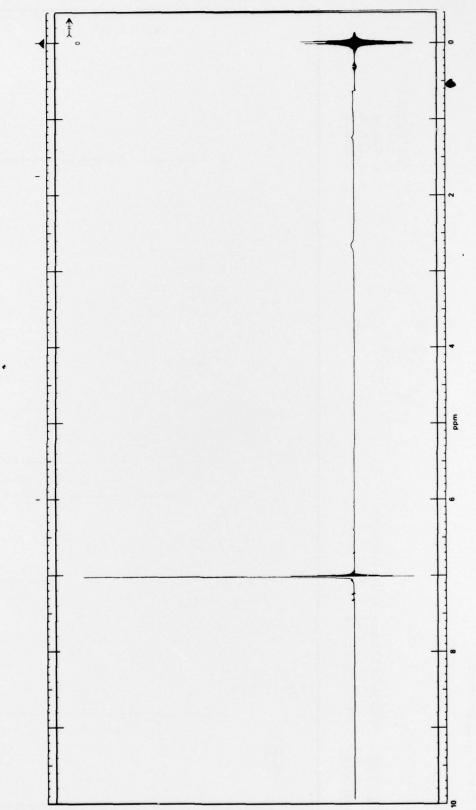




Spectrum 44 - Polyazelaic polyanhydride (Emery 9872 PAPA); solvent: CDC13







Spectrum 45 - Maleic anhydride (Fisher A-168); solvent: CDC13

Recrystallized from benzene

75% Acetone x

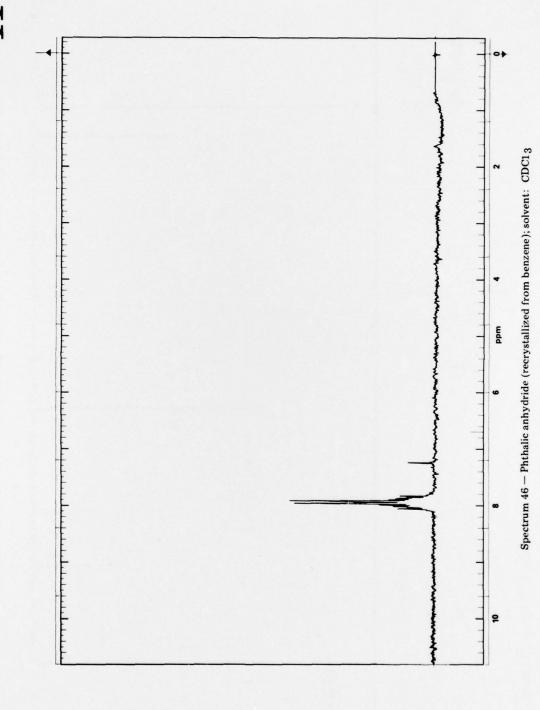
Solvent:

Source:

NRL

Assignments: a 125.5 b 131.6 c 136.4 d 163.2

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Tetrahydrophthalic Anhydride

The total Control State Section 1 St. Actor 1 16 comme

Assignments: a 26.2 b 39.4 c 125.3 d 175.3



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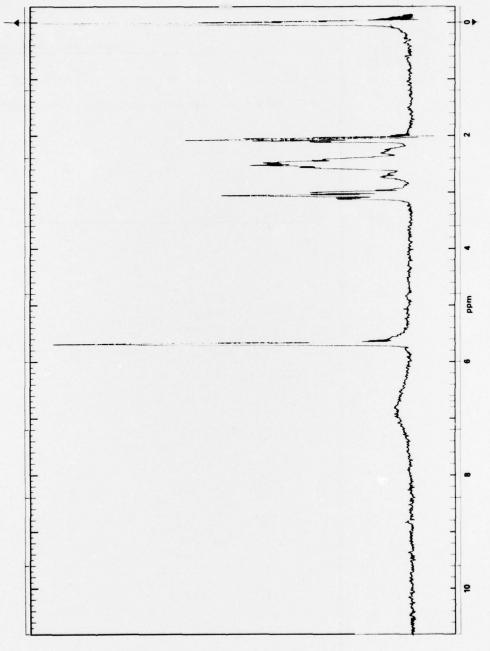
50% Acetone x

Solvent:

Source: Eastman #5724

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Spectrum 47- Tetrahydrophthalic anhydride (Eastman 5724); solvent: acetone-d6

Hexahydrophthalic Anhydride

Assignments:
a 21.4
b 23.1
c 39.8
d 172.1

50% CHC1₃× Solvent:

Miller-Stephenson Chemical Co.

Source:

NAN

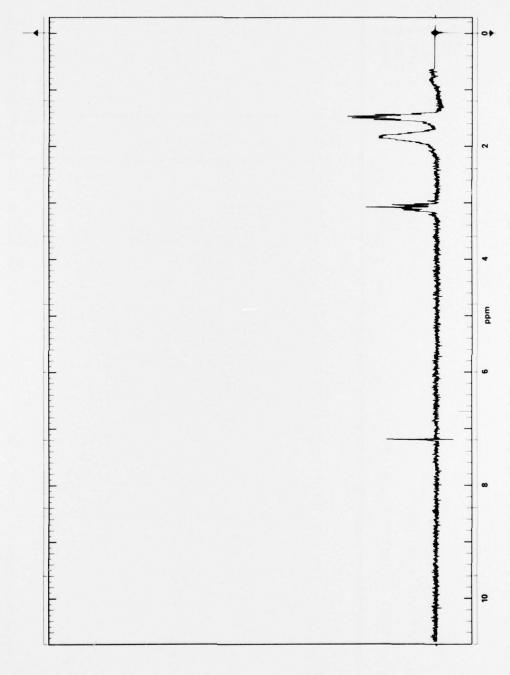
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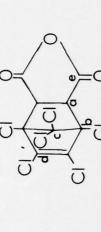
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Spectrum 48 — Hexahydrophthalic anhydride (Miller-Stephenson Chemical Co.); solvent: CDC13

1,4,5,6,7,7-Hexachloro-5-Norbornene-2,3-Dicarboxylic Anhydride



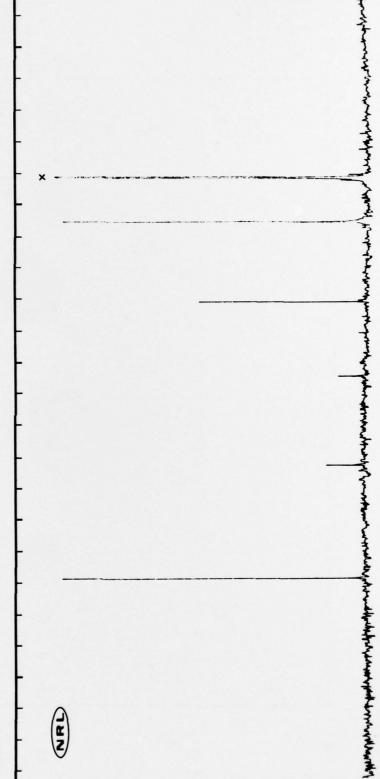
Assignments:
a 55.9
b 81.2
c 104.8
d 133.0
e 169.2

DMSO (saturated solution) x

Solvent: Source:

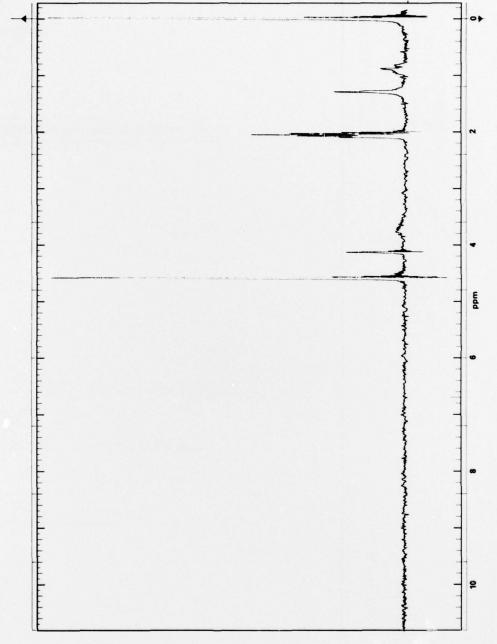
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Aldrich # 10,326-8



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Spectrum 49 - 1,4,5,6,7,7-Hexachloro-5-norbornene-2,3-dicarboxylic anhydride (Aldrich 10,326-8); solvent: acetone-de

g 127-148 h 170-173 £ 127-148 Assignments:
a 15-16.5
b 40-55
c 40-55
d 40-55

e 40-55 See Appendix for complete line listing.

NAC

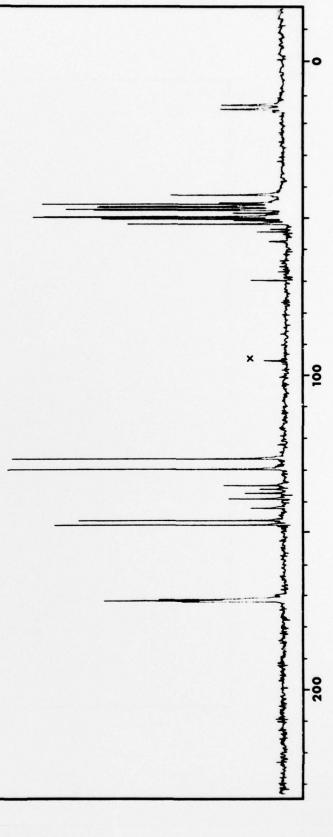
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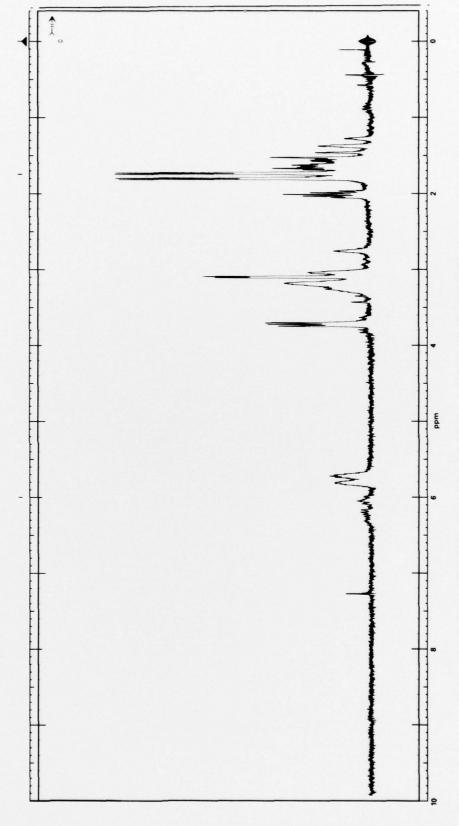
10% CC1<sub>4</sub>

Solvent:

CIBA 906

Source:





Spectrum 50 - Methyl-4-endo-methylene tetrahydrophthalic anhydride (CIBA 906); solvent: acetone-de